

# The Chemical Age

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**NOTICES:**—All communications relating to editorial matter should be addressed to the Editor, who will be pleased to consider articles or contributions dealing with modern chemical developments or suggestions bearing upon the advancement of the chemical industry in this country. Communications relating to advertisements or general matters should be addressed to the Manager.

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1930

## Good-bye to a Black Year

EVERYBODY will be glad to see the end of 1930. It has been a black year for nearly every class of industry, for the chemical industry no less than for others, and not for this country alone, but for the world in general. For a month or two it promised well for overseas chemical trade. Then progress was arrested and changed into retreat; towards the end of the year there has been a steady descent. The most one can hope for is that the lowest point of the curve has been reached and that the next change will be one for the better.

The statistics of overseas trade during the eleven months ended November 30 tell a very plain story. In the whole list of classified industries only one records an increase in exports, namely, vehicles (including locomotives, ships, and aircraft). The biggest

decrease is in cotton yarns and manufactures, nearly forty-two millions, with woollen and worsted goods and iron and steel next with decreases of over 14 millions each. The decrease in chemicals, drugs, dyes, and colours is small relatively to those in other industries, yet it amounts to the considerable figure of £3,600,839 compared with 1929, and of £2,936,624 compared with 1928. There is, however, this satisfactory feature—that imports have declined nearly in the same proportion—showing at least that British goods are not being replaced by foreign—and that there is still an enormous excess of chemical exports over chemical imports—£20,447,167 against £12,430,903. In this relative aspect the industry's position is sound, and when the turn comes—as of course it must, and as it always has done—then the advance will be in the right proportions. It is something at least to have maintained the ratio of exports to imports at such a good level.

In the striking series of articles we publish this week on the chemical experiences of the year, writers capable of speaking with knowledge and authority on the various branches offer their views as to the causes that have produced the present situation, and there is some desirably plain speaking. In particular, one may recommend attention to the considered terms in which Sir William Alexander, M.P., discusses the national situation, and to the remedies he suggests. One feature of the present depression—which may have a sort of doubtful consolation for some—is that it is world-wide. It has descended first on one nation, then on another, very much as a fog does, until—nations being so linked up by international finance and business—all are involved.

If for a moment we limit our view to this country alone and look for the causes, we should say that we have never yet recovered from the disastrous effects of the coal strike of a few years ago. The ill-balanced fanatics who proudly watched the coal industry and the industries dependent on it being brought to a standstill inflicted a deadly blow to British industry and provided untold suffering for thousands of work-people. Then we have had financial crashes following on a series of large scale swindling operations never before equalled. How much the present situation is due to the class of clever criminals who are now undergoing penal servitude it would be difficult exactly to say, but there can be no doubt that the shattering of credit on such a large scale and the heavy losses in which honest trading firms and investors were involved represent a considerable factor. One slight compensation is the public determination—in the case of large-scale or small-scale operations of this class—to bring the guilty parties to justice and inflict penalties that will stamp such practices out, and insist on the restoration of honest standards.

Looking back on the chemical events of the year, there are comparatively few outstanding features. The most exciting matter has been the controversy over the Dyestuffs Act with which the year closes. The Government have handled this matter about as unintelligently and clumsily as it could have been handled, and as a result of their surrender to the House of Lords, the Dyestuffs Act is to be continued for another twelve months—a valuable and a very reasonable concession to those firms who have restored this lost industry to its present position. The chemical industry inevitably follows the course of general industry, and in several cases—fertilisers may be mentioned as an example—output has had to be restricted in order to preserve some sort of ratio to consumption. The difficulty in such cases is that costly plant has to lie idle for the time being, and that people who have risked much in industrial enterprises go without reward. In spite of these unfavourable conditions, research and industrial enterprise has been fairly active in many directions—of which the solvent industry is perhaps the most notable example—and it is satisfactory to know that when conditions begin to improve this country is as well placed as any to take prompt advantage of the change.

To the many distinguished contributors who have so generously co-operated in making this Annual Review Number the representative issue it is, we offer once more our sincerest thanks and appreciation. To our advertisers and readers at home and overseas—who have bravely stood by their industries and resolved to see the temporary troubles through—we offer our congratulations on their typical British pluck and the hope that it may soon bring its reward. To all our friends throughout the great chemical industry we send in all sincerity the old-fashioned seasonable greeting of a Merry Christmas and a New Year of returning prosperity.

### Above the Salt

SURELY chemists are entitled to sit above the salt; we have made so much use of it, know so much about it, based so many of our theories on its behaviour. A good many years ago a wrathful letter appeared in the papers denouncing the infamies practised at a Royal Society soirée where a bulldog placidly spent the evening with its feet immersed in a mixture "of that corrosive metal sodium and the poisonous gas chlorine" whilst electric shocks were sent through its body. Most of us have since submitted to this treatment without complaint; truly a little knowledge is a dangerous thing. Yet is it not wonderful that the salt we eat and look upon as one of the most harmless and neutral of materials, which, in the form of refined salt is one of the very purest of all known substances, should be capable of re-solution by a relatively small expenditure of electric energy into two such diverse, such reactive substances?

The story of salt should be written in such a way that every adult could understand it; there can be no better introduction to the achievements and wonders of chemistry. What a grand chain can be made of the happenings when a chemist decides to play with sodium chloride: he electrolyses it to start with, and

converts the chlorine into bleaching powder or into a chloride of sulphur which somehow helps to make acetic anhydride and that, in turn, silk stockings. Meanwhile the sodium produced at the other pole of the cell easily becomes cyanide, and is used on the far Rand to extract gold, maybe to pay for the stockings. Where else is there such romance?

And chlorine, too, has ways of its own, horrible ones in war-time. As Ellwood Hendrick once put it, "the halogens have any number of Celtic traits; green chlorine and its cousin fluorine are as full as tricks and potentialities of danger as any Irish lass who ever lived." Unscientific language perhaps, but more appealing than any page in an inorganic text book.

The purity and the utility of salt has made the name a synonym for goodness.

There is romance and history, too, in the winning of salt; the old Scotch abbeys were endowed with salt-pans as early as the twelfth century, and within memory the ruins of salt pans were scattered along the coast of Fife, the industry being destroyed by the repeal of the duties. We have all read about the wonders of rock salt mines, though to-day the product is brought to the surface by the more prosaic method of pumping saturated brine. We think of salt also, and perhaps of other things, when reference is made to Salt Lake City or to the Dead Sea. As chemists, we should then be pondering over the forces which hold chlorine and sodium so firmly together, the structure of the molecule, the atom, the electron and all those visions of the ultimate structure of matter which the spectacles of scientific discovery have enabled us to see.

Much as we already know, the salt has not yet lost its savour for future investigation.

### Books Received

THE TEXTILE RECORDER YEAR BOOK. 1931. Manchester: John Heywood, Ltd. Pp. 898. 7s. 6d.  
 " ARE CHEMICAL PLANT EXHIBITIONS OF ANY USE ? " London: British Chemical Plant Manufacturers' Association. Pp. 36.  
 THE RELATIVE VALUES OF COD LIVER OILS FROM VARIOUS SOURCES. By Professor J. C. Drummond and Professor T. P. Hilditch. E.M.B. 35. London: H.M. Stationery Office. Pp. 129. 1s.  
 TEXTBOOK OF QUANTITATIVE ANALYSIS. By William Thomas Hall. London: Chapman and Hall, Ltd. Pp. 264. 12s. 6d.

### The Calendar

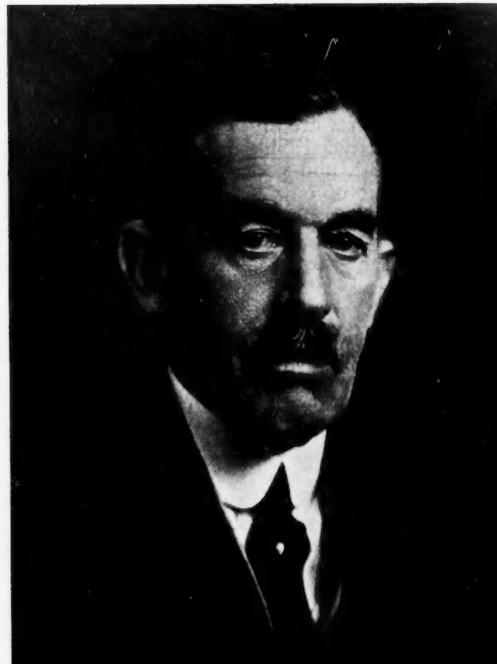
| Jan.      |  |   |  |
|-----------|--|---|--|
| 5         | Society of Chemical Industry (London Section: "Strychnine.") Professor R. Robinson. 8 p.m.                       | Burlington House, Piccadilly, London.                                 |  |
| 5         | Institute of Metals (Birmingham Section): "Tubes." 7 p.m.  | Chamber of Commerce, New Street, Birmingham.                          |  |
| 6         | Optical Society: Twenty-first Annual Exhibition.   | Imperial College of Science and Technology, South Kensington, London. |  |
| 6, 7, & 8 | Electroplaters' and Depositors' Technical Society: "Modern Metal Cleaning." 8.15 p.m.                            | Northampton Polytechnic Institute, London.                            |  |
| 7         | Institute of Metals (London Section): "Extrusion." R. Genders. 7.30 p.m.   | 83, Pall Mall, London, S.W.   |  |
| 8         | Society of Chemical Industry (Bristol Section): "Electrolysis of Fused Zinc Chloride." E. A. Ashcroft. 7.30 p.m. | University, Bristol.  |  |
| 8         | Oil and Colour Chemists' Association: "Solvents." T. H. Durrans. 7.30 p.m.                                       | 30, Russell Square, London.   |  |



*Sir William Alexander, M.P. for Glasgow (Central), and a well-known figure in British and International Chemical Industry.*



*Sir H. Sutcliffe-Smith, chairman, Colour Users' Association.*



*Sir David Milne-Watson, Governor of the Gas, Light and Coke Co.*



*Dr. E. F. Armstrong, F.R.S., chairman of the Association of British Chemical Manufacturers.*

## British Industry in 1930

By Brig.-Gen. Sir Wm. Alexander, K.B.E., C.B., C.M.G., D.S.O., M.P.

*The distinguished author of this article discusses not only the chemical situation, but the general condition of industry, with a direct knowledge and frankness that will be warmly appreciated.*

TEN years have elapsed since I first responded to the request of the Editor of THE CHEMICAL AGE for a review of the state and progress of the British Chemical Industry during the previous year. At first largely concerned with the Dyestuffs Industry, these reviews have in later years taken a wider survey. They have attempted to analyse the essential conditions of progress, to emphasise the importance of new industries built on research, and to point out the dangers of political interference. The experience and knowledge acquired in this way are reinforced by the intimate and practical experience of a lifetime spent in the chemical industry.

This background gives added force and definition to the statement that on no occasion during the past quarter of a century could one have sat down to write a review on a great basic industry with less feeling of satisfaction than when considering the achievements and results of the past year.

### A Gloomy and Disappointing Picture

As in the case of practically all our basic and manufacturing industries, a review of the Chemical Industry for 1930 presents a gloomy and disappointing picture, and none engaged in chemicals, solvents and allied products will regret its passing, while hoping that we have touched bottom and that 1931 will bring some reassuring, even if only slow and gradual, return to more normal conditions. This will depend on a number of factors, but, so far as Britain is concerned, not the least important will be the incidence of politics.

Every manufacturing industry, during good or bad times, has its direct or indirect effect on the consumption of chemicals and solvents. The state of the Chemical Trade is the barometer of trade in general, and especially so since the war, when chemical discoveries and new industries arising therefrom have been the one bright spot in the reconstruction of world peace-time production and trade.

Following the financial crisis which started towards the end of 1929 with the debacle in stocks and shares, a worldwide slump in trade was the inevitable result. It has increased in intensity throughout 1930 and the Chemical Industry has borne its full share of commercial misfortune, as shown by home and export statistics, and confirmed by the increase in unemployment of 8 per cent. over the year.

Efforts have been made, especially by political parties, to lay the entire blame of our trade depression and staggering unemployment figures on the conditions resulting from stock and share depreciation on Wall Street and Throgmorton Street, with the consequent loss of capital, credit and confidence. These have been contributive, but so far as Britain is concerned, they have not been the sole or even the prime movers.

### Political Factors

I shall hope to show by a few examples before closing that more important political factors, resulting from ignorance and maladministration over the past 10 years by succeeding Governments, have largely contributed to and accentuated the ill-fortunes which have handicapped our manufacturing industries in developing peace-time trade, have driven our best industrial administrators and leaders almost to despair, and, worse still, have augmented continuously the mass of unemployed, dependent entirely on profitable industry for decent livelihood and existence, while the nation is passing from the healthy position of

paying its way out of revenue to the disastrous one of paying it out of capital.

The flywheel of British prosperity is being slowed down for want of commercial experience in high places, where the merest amateurs, untrained in commercial practice, are the first called upon as politicians to advise, guide and legislate, with grave results, on questions of national commercial importance where the most astute commercial brains in the country should be consulted.

To-day every industrial country has more plant capacity than is required for internal production requirements, and is looking and striving to clear surpluses overseas, even at prices which contribute merely to the relief of overhead charges. Britain is in a similar position so far as capacity is concerned, but she has not even the security of her own home markets, and is debarred from exporting through tariff walls abroad.

Curtailment of consumption has led to accumulation of stocks, and, during a time of financial stress, these must be turned into cash wherever markets can be found, irrespective of economic values.

Consequently, commodities of all kinds have reached price levels in international competition for business, which are seriously affecting balance sheets and wiping out the profits on which the country relies for taxation. The result will be seen in the coming National Balance Sheet, when the disaster of spending more than the earnings of a nation can meet will be realised in its true light.

### Reasons for Diminished Output

Coming back to the subject under review, a few instances of the reasons for diminished output, which are typical of all, will be instructive.

The market for heavy chemicals has been and continues to be seriously influenced by the curtailed demand for textile goods where the bleaching, dyeing, printing and finishing industries are all large consumers.

The new Artificial Silk Industry, which is not only one of the great, and perhaps the greatest, employers of labour per unit of output, but the greatest market for chemicals and solvents, was shot to pieces early in the year by the Government's proclamations regarding safeguarding duties. It has never yet recovered from this interference, and millions of money invested in new concerns have as a consequence been lost to British investors.

This is one typical example of a new and growing industry of vast importance to the chemical, engineering and other industries, and contributive materially to the employment of large masses of labour being sacrificed through party politics, while large capital expenditure made for the purpose of feeding this industry is to-day lying dormant.

Reduced demand through decreased spending power in other new industries, like artificial leather and cellulose lacquers, has affected adversely the sale of solvents and plasticisers, another of the newer industries recently established in this country.

The depression in agriculture through foreign dumping has restricted seriously the consumption of the synthetic fertilisers for the production of which millions of money have been invested.

### A Missed Opportunity

We have recently had much discussion and debate on the suspension of the Dyestuffs (Import Regulation) Act. If proof of political damage and obstruction were further required, the history of Government legislation in regard

to this Act provides a convincing and lamentable record which has not been fully disclosed in debate.

The bargaining power of this piece of protection gave to those in charge of the British Dyestuffs Corporation in 1924 an opportunity of concluding a sound commercial treaty with the German Dyestuffs Industry. An agreement was drafted, the protocol of which was actually initialled, whereby the following benefits would have been definitely conceded to the British Industry :—

1. A share of the world trade, which would have kept the British factories fully employed.
2. A price regulation which would not have penalised British dyestuffs users, either in choice of their source of supply or in prices.
3. Free access to all the patents and processes evolved by the Germans in over 50 years' experience and a guarantee that by exchange of chemists British products would equal in quality the corresponding German ones.

The Government, and a Conservative one at that, by reason of its powers of veto as a shareholder and through representations of vested interests, actually vetoed this transaction, which, had it been concluded, would have eliminated by now any necessity for the Act and rendered unnecessary any discussion as to its continuance.

Legislation of this kind in connection with purely commercial questions has stalled and retarded the strenuous efforts of experienced commercial men to bring back prosperity to trade and industry, the main bulwarks of national success and welfare.

#### National Economy

To these follies we have to add the failure of successive Governments to achieve national economy. We have to record the squandering of millions on doles, contributing nothing in return, extravagant expenditure on national services worked by an inefficient administration, political first and commercial last, which cannot and will not see the red light, so that already the country's capital and wealth are fast being squandered and taxation is being

raised to a point where our industries can no longer compete in the race for supremacy.

Is it to be wondered at that :—

1. We have plant and machinery far in excess of the spending power of the population.
2. That stocks have accumulated to great superabundance and are being turned into cash at heavy losses.
3. That factories are running on short time through uneconomic costs, largely due to overpowering taxation, while unemployment is at a colossal figure and still rising.
4. That thousands of families with limited means have domiciled themselves abroad, mostly in Europe, to avoid taxation, with the consequent loss of millions of money per annum which should have been circulating at home.

#### One Solution Only

There is one solution and one only, and there are signs that the general public are waking up to the fact. The solution is that all questions and problems of a commercial nature affecting national progress and welfare should be removed from the influence and inept handling of the politicians who are usually, in our antiquated system of administration, round pegs in square holes.

The House of Commons attracts the best legal brains, which is to the good, so far as purely legal work and bill drafting are concerned, but, unfortunately, the House does not attract in sufficient numbers what the nation most needs there, namely, the best and most astute and experienced commercial brains. Until it does, a committee or commission of, say, three of our greatest commercial and financial men of long experience, free from Parliamentary ties and political bias, should be set up and given the largest authority possible to consider, on national merits, all commercial problems and advise the politicians what action would be in the best interests of the nation. Then, and only then, shall we make some real progress towards running the Empire at a profit.

## A Review of British Chemical Industry in 1930

By J. Davidson Pratt, O.B.E., M.A., B.Sc., F.I.C.

*His position as general manager of the Association of British Chemical Manufacturers gives our valued contributor, Mr. Davidson Pratt, exceptional means of accurately estimating the present conditions in British chemical industry. While frankly recognising the effects of world-wide depression, the article notes a number of favourable points, and indicates two practical steps that might be taken to improve matters—(1) the purchase of British chemical products in place of those at present imported, and (2) the capture of the Colonial trade that now goes to foreign countries.*

In commenting on the progress of the British Chemical Industry in 1930, one is handicapped by the lack of data for the last two months of the year, since at the time of writing the import and export data are available only to the end of October. Nevertheless, it is possible to draw fairly accurate deductions as to how 1930 will compare as a whole with 1929. The chemical industry is the great purveyor of raw materials for every industrial activity, including agriculture, and consequently its position is a fairly accurate reflection of a country's industrial prosperity as a whole. Needless to say, our chemical production during the past year has suffered from the curtailment of numerous outlets for its products owing to the serious depression in many of our old-established industries and in agriculture, while the world-wide nature of the slump is reflected in our export trade.

#### General Progress

If we turn to the index of production figures issued quarterly by the Board of Trade, we find that the index (1924 being taken as 100) for the last quarter of 1929 was 111.5, while the final index for the whole of 1929 was 115.8. This shows a steady advance on 1927 and 1928. The figures for the first three quarters of 1930 have been 104.9, 101.3, and 106.7. Though these figures are all much lower than in 1929, the increase in the September over the June quarter gives some cause for hope that production is again on the up grade.

There is, however, a complication, in that this index includes the refining of petroleum—normally regarded as outside the chemical industry proper—in addition to the manufacture of heavy chemicals, explosives, dyes, ammonium sulphate, sulphuric acid, soap and matches; and the data do not permit of the elimination of the effect of the petroleum industry.

Unemployment figures published by the Ministry of Labour make less pleasing reading, but there is again a complication in that the term "chemical industry" is here used in a more restricted sense, and does not include the industries making soap, artificial silk, paint, glue, oil and matches. The estimated number of insured persons employed in the industry between the ages of 16 and 64 was 105,340 for the last two months of 1929, and the first five months of 1930. This has dropped during the last five months to 102,930. The percentage unemployed has steadily increased from 6.8 at the end of 1929 to 14.9 at the end of October, 1930, an increase of 8.1 per cent., the actual number unemployed being 15,344. These figures are very disturbing and tend to counteract the optimistic expectations derived from the index of production.

The export and import figures do not provide much comfort. The 1929 exports of chemicals, drugs, dyes and colours were valued at just over £26,500,000, but the 1930 exports are likely to be about 15 per cent. less. Imports have also decreased, the estimated reduction being nearly 20 per cent. on the 1929 total of nearly £17,000,000. Re-exports, which in

1929 were approximately £950,000, so far show a slight increase. The subsequent paragraphs will indicate the directions in which these decreases have mainly occurred.

An examination of the detailed import figures shows that many of the products can or could be supplied here in quantities sufficient to meet all demands and at world prices. The fact that they still come into the country is due in some cases to dumping; in others it is due to the preference which some British buyers still show for foreign products, even when there is little or no economic advantage to be gained by buying abroad. This is a reprehensible state of affairs; such buyers are not doing their duty by their country, and are, in fact, injuring their own interests in view of the resulting unemployment and the repercussive effect on taxation which falls on them as on others.

#### The Sulphuric Acid Barometer

Although the production of sulphuric acid is no longer an accurate barometer of our general industrial condition, the output figures are nevertheless worth careful study. Authoritative figures giving the amount of acid manufactured, and the percentage of plant capacity in operation in the United Kingdom and Ireland, are only available for the first nine months of 1930, and the following table shows how these compare with past years:—

Output of Sulphuric Acid and Plant in Operation.

| Year.                      | Tons  |          | Percentage<br>100 per cent. of<br>$H_2SO_4$ Capacity. |
|----------------------------|-------|----------|---|
|                            | Oleum | Imported |   |
| 1927—last six months       | ...   | ...      | 434,000 62.0  |
| 1928—first six months      | ...   | ...      | 457,000 66.4  |
| 1928—last six months       | ...   | ...      | 471,000 67.3  |
| 1929—first six months      | ...   | ...      | 488,000 71.3  |
| 1929—last six months       | ...   | ...      | 479,000 70.3  |
| 1930—first six months      | ...   | ...      | 463,000 68.3  |
| 1930—July—August—September | ...   | ...      | 192,000 56.9  |

It will be observed that the year 1930 will show a very marked decrease in the amount of sulphuric acid manufactured, whereas in previous years a steady increase was recorded. This, unfortunately, is only a reflection of universal trade conditions; it is not due to a decrease in the demand for superphosphate, the production of which actually increased. The data relating to the months July, August and September, 1930, show that only 56.9 per cent. of the plant capacity was in operation; this represents a drop of 15.8 per cent. on the corresponding figure for the same period of 1929, and the indications for the last three months of this year are similarly depressing.

As regards raw materials, figures of consumption are only available for the first six months of 1930 and are contained in the following tables:—

Total Acid made and Proportions of Raw Materials used.

| Year            | 100% $H_2SO_4$ | Percentage Made from |         | Zinc<br>Concen-<br>trates |
|-----------------|----------------|----------------------|---------|---------------------------|
|                 |                | Oleum, in<br>tons of | Pyrites |                           |
| 1928            | 928,000        | 44.18                | 0.81    | 25.64 20.12 9.25          |
| 1929            | 967,000        | 47.54                | 0.81    | 24.62 17.71 9.32          |
| 1930 (6 months) | 463,000        | 49.40                | 0.64    | 25.35 14.77 9.84          |

Output of Acid and Consumption of Raw Materials in Tons.

| Year            | 100 per cent. $H_2SO_4$ |             | Pyrites | Spent<br>Oxide | Sulphur | Zinc<br>Ores   |
|-----------------|-------------------------|-------------|---------|----------------|---------|----------------|
|                 | Acid and<br>Oleum       | Net<br>Acid |         |                |         |                |
| 1928            | 928,000                 | 782,000     | 146,000 | 329,000        | 174,000 | 58,300 116,400 |
| 1929            | 967,000                 | 808,500     | 158,500 | 363,500        | 174,500 | 52,700 115,600 |
| 1930 (6 months) | 463,000                 | 384,500     | 78,500  | 178,600        | 85,200  | 21,000 38,000  |

(The figures for 1921-1927 inclusive will be found in last year's report.)

It will be seen from the above that pyrites continues to keep pride of place, and, in fact, has distinctly gained ground of recent years. This is caused by the decrease in the consumption of brimstone, which in 1927 represented 24.6 per cent. of the total acid manufactured, and now represents less than 15 per cent. It is understood that this diminution is due primarily to the advance in the price of Sicilian sulphur, and to the great uncertainty which exists regarding future supplies from that source.

The quantity of acid manufactured from spent oxide remains substantially constant at a quarter of the total, and it should be borne in mind that at the present time the consumption of spent oxide is probably slightly in excess of production, with the result that prices have hardened con-

siderably, and stocks both at gasworks and at acid manufacturers' works are decreasing.

With regard to the quantity of acid manufactured from the calcination of zinc concentrates, it will be seen that the upward trend continues, and the prospects indicate that the maximum has not yet been reached.

With regard to prices during the current year, cost of production has risen, due to the increase in the average cost of sulphur in all forms and to the decreased output, but there has not been any attempt to meet this by a general change in prices, which are, in fact, slightly lower than those maintaining during 1929. Whether these prices will be maintained during the coming year is a matter for conjecture, but it is certain that, in view of the great increase in the cost of brimstone, some increase in price would be justifiable in the case of arsenic free acid.

#### Heavy Chemicals

The home trade in alkali products has suffered as a result of the decreased demand, particularly for the manufacture of rayon. The other consuming industries, such as glass, soap, and paper, have maintained their demand fairly well in spite of the general trade depression. There has also been a decline in the export trade. The 1929 exports of sodium compounds were about £3,864,000, but for 1930 the value is expected to be about 10 per cent. lower.

Owing to slackness in the galvanising trade, there has been a diminution in the demand for hydrochloric acid. The decrease in the demand for chlorine products has been almost entirely due to the depression in the cellulose acetate silk section of the rayon industry; the export trade has been well maintained in spite of severe competition in certain overseas markets. The use of chlorine for water purification has increased. Output of citrates and tartrates has shown some reduction owing to depressed conditions at home and abroad, while the export trade has declined. Prices have fallen owing to the acute competition for business and are now actually below pre-war in the case of citric and tartaric acids and cream of tartar.

Borax and boric acid production has been maintained, though trade has lately shown some falling off. These chemicals are, however, finding new uses in a variety of industries, such as in the manufacture of bottles and other glass containers; the production of tough glass of the type required for electrical insulation, lighting and cooking; the melting of alloys; the fabrication of stainless steels; copper welding; the manufacture of adhesives; textile finishing and the fireproofing of fabrics. Prices have advanced somewhat, but are still much below the pre-war level. The difficulties of over-production are being met by stimulating consumption and finding new outlets, while economies are being obtained by concentrations to obtain bigger units of production and the use of borate of soda for borate of lime in the manufacture of borax.

Hydrogen peroxide is an example of the prevailing tendency for production to outrun consumption. While the old establishments are well equipped and up-to-date technically, some of the newer concerns abroad have still to overcome their initial difficulties and may have hard work to establish a satisfactory position. Trade in this country has been well maintained in spite of the textile depression and compares favourably with that in the more important European markets. The fall in price has continued, being about 12½ per cent. during the past year, till it is very little higher than pre-war. These reductions have been possible, not so much because of the decreased cost of raw materials, but because of increased efficiency in manufacture and a larger production, resulting from extended use at the lower price level.

Speaking generally, the production of heavy and industrial chemicals has suffered from the general depression, except in cases where new outlets for a product have been found. In certain lines the home trade has been disturbed by the sale of Russian material at uneconomic prices, though the chemical industry has not suffered so much from such dumping as other industries have. Price levels have, on the whole, been maintained, though the effect of the reduced production has had and will have a tendency to make prices rise.

Exports of general chemicals (that is, chemicals other than fertilisers, medicines, dyes, coal-tar products, and painters' materials), were close on £11,000,000 in 1929, but in 1930 are likely to be down by some 10 per cent., due to decreases in practically every item. The 1929 imports of general chemicals

were valued at £9,300,000. The 1930 figures are likely to show a drop of nearly 30 per cent., owing to reduced imports of practically every material enumerated.

#### Fertilisers

The subject of fertilisers can be briefly dismissed, as it will be discussed in more detail elsewhere. The annual report of the British Sulphate of Ammonia Federation, recently summarised in this journal, gives an excellent review of the world position in regard to nitrogen fertilisers. Owing to the general agricultural depression, the steady increase which has taken place in the use of nitrogen manures in recent years was not maintained throughout the world, except in the case of Spain and Portugal. Synthetic nitrogen gained ground, however, at the expense of Chile nitrate.

Owing to production outrunning consumption, the position during 1930 was at one time serious, but fortunately the European producers were able to come to a one-year agreement which will serve to stabilise the industry until a more permanent basis of co-operation is secured. The production of sulphate of ammonia in the United Kingdom rose from 645,042 tons in the fertiliser year 1928-29 to 865,878 tons in 1929-30, while the consumption for home agricultural purposes declined from 186,710 to 183,516 tons; exports for the fertiliser years 1928-29 and 1929-30 show an increase from 428,437 to 634,405 tons. When the calendar year 1930 is compared with 1929, there is an estimated increase of 14 per cent. in weight, but a decrease of 5 per cent. in value on a tonnage of 587,476, valued at £5,600,000; there has been a 50 per cent. drop in the case of Japan which is more than counterbalanced as far as weight is concerned by increases to Spain and other countries. Imports of Chilean nitrate are down by nearly 50 per cent.

The production of superphosphate rose from 556,400 tons in the fertiliser year 1928-29 to 575,600 tons in 1929-30, while imports are likely to be down in 1930 by at least 25 per cent. in value as compared with 1929.

In considering the fertiliser position, it is discouraging to feel that the industry has not reaped the expected benefits from the technical achievements of which it has shown itself capable, owing to the lack of adequate demand from the agricultural interests.

#### Explosives

The decline in the home market for explosives is due mainly to the decreased coal output, although the reduction in tin mining has had some effect. In the export trade the full force of the world depression has not been experienced, but the unsettled conditions in various South American Republics have been detrimental.

#### Coal Tar Products

In the case of coal tar products conditions have been difficult throughout the whole of this year, and prices generally have shown a decline. Exports of creosote oil to the United States have been below the figure anticipated, and stocks are correspondingly large. The National Creosote Committee is working on the problem of new outlets.

The concerted efforts made to stabilise the price of pitch have been so far successful, and the value has been maintained throughout the year at 45s. to 47s. per ton f.o.b. Sales have been fairly satisfactory, and it is expected that the stock position at the end of the year will not be worse than at the end of last year.

Fortunately, the demand for road tar has been very good during the summer, both for export to the continent and for the home market, and this has resulted in the production of less pitch and oil than would otherwise have been the case. Although the increase in the quantity of road tar used is very satisfactory, the price level, unfortunately, still continues low.

Tar spirits—*i.e.*, benzole, toluole, etc.—have been somewhat quieter, and values have declined in sympathy with the reductions that have taken place in petroleum spirit. Carbolic acid has been quiet since the beginning of the year and values have declined. Pyridine has been dull. Naphthalene has been in fair demand throughout the year, but competition from the continent has been keen, and sales made have been at about the equivalent of creosote value.

The 1929 exports of coal-tar products were valued at £1,730,000, while the 1930 figures are likely to show a drop of nearly 30 per cent.; the greatest decrease has occurred with tar oil and creosote oil, with substantial reductions for benzole and carbolic acid. The imports, which normally represent

little more than a quarter of the exports, are also likely to be down about 30 per cent.

Though a great deal of work is being done on the technology of coal tar, and in particular low-temperature tar, there are no important advances to record, though promising results are said to have been obtained in various quarters in the hydrogenation and cracking of tar and its fractions with a view to the production of motor spirit. None of these has yet reached the commercial stage, and the problem of the economic utilisation of low-temperature tar remains unsolved.

#### Wood Distillation

The year 1930 has not been one of great encouragement to those engaged in the wood distillation industry, and few firms have been able to maintain their works operating at full plant capacity.

The continued depression in textiles, artificial silk, and metallurgical processes has had its reflex action on the demand for wood chemicals, and the price movements have been generally downwards. In addition to the more severe competition from continental sources, the trade has had to take into account the new factors of synthetic methyl alcohol and synthetic acetic acid, both of which are disturbing elements from the wood distillers' point of view. At the same time those engaged in the industry have been spending money on research and up-to-date plant in the endeavour to maintain as high a standard of quality and economy as possible.

The Suida process for acetic acid is being developed in this country, and several new solvents have been put on the market and are finding an outlet.

Wood distillers, like many other manufacturers, are hoping in the future for some protection from the tremendous competition of imported products coming from countries where production is not hampered, as it is here, by high wages and taxation.

#### Dyestuffs

Although no figures are yet available for the production of dyestuffs and intermediates in this country during the year 1930, there is good reason to believe that they will be well up to the 1929 output, in spite of the depression in the dyeing and printing trades, which has been accentuated during the latter half of the year under review, and in spite of the disturbing effect of the Government decision to let the Dyestuffs Act lapse on January 14, 1931.\*

The total production in 1929 was the record one of 55,785,032 lb., showing an increase over the previous year of nearly 5,000,000 lb. One of the most pleasing features in these returns is the continued advance in the output of the vat colours generally, including indigo, the comparative figures being 11,287,342 lb. in 1928 and 14,683,701 in 1929. Included in the latter figure is a total of 2,659,300 lb. for vat colours other than indigo, and, as several additions to this series of fast colours have been made recently, it is expected that the production of these colours for 1930 will be a record one.

As regards the trade in dyestuffs, the slump in the Lancashire and Yorkshire textile industries, coupled with the Indian boycott, has been reflected in the lessened demand for certain classes of colour. On the other hand, trade in the fast colours, both in printed and dyed goods, has been fairly well maintained.

This state of affairs does not appear to be confined to this country alone, but has its counterpart in practically every quarter of the world where similar work is carried on.

The exports of dyes and intermediates in 1929 reached the record value of £1,087,000. Unfortunately, the steady increase in the export trade is unlikely to be maintained, owing largely to difficulties in the Chinese and Indian markets, and a small reduction has to be faced for 1930. Even so, the figures are still expected to be well ahead of those for 1928. As regards imports, these represent specialities which are not yet made in this country, and comprise nearly 2,000 different brands, so that the greater part of the imports consists of quite small amounts of high-priced materials. The value in 1929 was £1,120,000, and is not expected to show much change in 1930.

There has been no outstanding alteration in the price of dyestuffs during the current year, but the tendency has been in the direction of a steady lowering of prices of dyewares generally.

\* This decision was later reversed.

Important additions to the range of fast colours, both for wool and cotton, have been made by British manufacturers, thereby helping to fill up the gaps in the existing series, thus obviating the necessity of importation under licence.

#### Painters' Colours and Materials

The production of painters' colours and materials has been fairly well maintained, and competition in many lines has been keen. Exports have decreased, as in many other lines, the estimated value in 1930 being of the order of 15 per cent. below the 1929 total of over £4,000,000. Imports are also down about 5 per cent.

#### Fine Chemicals

The production data for fine chemicals for 1930 will not be available for some months yet, but there is every indication that the total volume will exceed that in 1929. This is due mainly to new manufacturing developments, such as those of Imperial Chemical Industries, Ltd., for acetic acid and rubber accelerators, and of British Industrial Solvents, Ltd., for acetic acid, acetone, and butyl alcohol. These new activities will go a long way to reduce the imports of fine chemicals liable to Key Industry Duty to which reference is made later.

In the case of medicinal products, the home trade has been slack, probably due in part to the fine summer in 1929 and the mild winter; buyers have also been unwilling to purchase beyond their immediate requirements in view of the possibility of a fall in prices. In the export markets trade has been very difficult indeed, for a variety of causes: the general trade depression has played a part; the Indian boycott has had a severe effect; the increase in the Australian tariffs has practically shut out certain lines entirely, such as bismuth salts; while there has been intense competition from continental makers, who have the benefit of lower wages and much less taxation. In 1929 the exports of drugs, medicines, and medicinal products, which had shown practically a steady advance since 1921, reached the value £3,300,000, but the 1930 figures will show a drop of over 10 per cent. for the above reasons. Imports for 1930 may be expected to decrease by about 15 per cent. on the 1929 value of £2,600,000.

There have been few price movements of importance to record, and generally speaking the tendency has been downward. In a few lines there has been an upward movement as a result of international agreements, which, having involved the limitation of production, have justified the maker in asking for a higher price. It is instructive to note, in connection with the operation of the Key Industry Duty, that in those cases where there is as yet no real British competition, the continental prices have been advanced. The research work of the industry has continued with unabated vigour and a number of new therapeutic products has been put on the market with valuable results in the treatment of disease, while the methods of manufacture have been improved in a great many cases with benefits as regards output, quality, and cost.

There is very little to say as regards photographic materials. Production has been steadily maintained, and we have held our own as regards our share of the world trade. The home market is affected by the weather conditions during the holiday season; it started strongly with the early fine weather, but it finished sooner and more abruptly than usual owing to the bad August and September. The export markets have suffered severely owing to the general depression, since photography is a luxury trade and very susceptible to such influences. Prices generally were on the 1929 level. There have been no developments in technique which have affected the use of photographic chemicals.

The essential oil industry has seen a number of important price changes. Oil of lemon has dropped steadily from 9s. 6d. to 3s. 6d. per lb., due primarily to a very heavy crop in Sicily, coupled with the effects of the general trade depression. The same is true of other Sicilian oils such as bergamot and orange, and higher prices are unlikely for some time. Owing to the tornado in Dominica, oil of limes has been scarce and prices have risen. This will be maintained for some time, as it takes four or five years before new trees are in full bearing. The price of clove oil has been very firm owing to the shortage of this year's crop, and the high price of cloves has made vanillin, produced therefrom, dear in comparison with that prepared from other materials. Oil aniseed was high in price

in the early part of the year, due to export difficulties arising from the disturbed state of China, but with recent easier shipping facilities and the change in the value of silver, the price has now fallen to a low level. Imports of essential oils which were valued at £1,300,000 in 1929 have fallen this year by some 20 per cent.

There is little to say on synthetic perfumes as the work has been devoted to the perfecting of existing processes in order to put them on a bigger scale and make them more competitive.

The position as regards solvents has steadily improved as a result of new manufactures, such as are indicated in the opening paragraph of this section; the demand in certain directions has, however, been affected in the last few months by the trade depression. The foreign trade has been influenced by the Indian boycott and Australian tariffs. The price level has declined slightly. Technical fine chemicals have suffered from the general depression as was to be expected.

The normal statistics issued by the Board of Trade for imports and exports do not contain the data necessary to show how the foreign trade in fine chemicals varies from year to year. Since imports of fine chemicals are subject to a Key Industry Duty, the Customs are required to keep special statistics to show the value of the yearly imports. Figures covering the years 1922-1929 inclusive, were published in Hansard in the early part of the year, and showed that the value of the Key Industry Duty chemicals had been steadily increasing during the past few years; the value of the retained imports in 1929 was £1,340,452 made up of £563,007 from other parts of the Empire which come in free, and of £777,445 from foreign countries which pay duty. These figures might give the impression to a casual observer that the Key Industry Duty was not fulfilling its purpose of developing the fine chemical industry. A detailed investigation of the figures has recently been made by the Association of British Chemical Manufacturers, which proves conclusively that this is not the case. A great many factors operate, such as lack of raw material and cheap power, patented processes, and under-declarations or dumping, but the main consideration is that new developments in certain industries such as artificial silk, lacquer, and rubber, have increased the demand for fine chemicals in a remarkable manner in the last few years, and the home industry, in spite of a great extension of its production, has not been able to keep pace with this demand. This is rapidly being rectified and the next few years should see a great diminution in these imports.

#### Conclusion

This record of 1930 makes disappointing reading, but it is better than might have been expected in the light of all the circumstances. The world-wide gloom which has fallen on practically every industrial activity shows signs of being dispelled at no distant date. The question which arises is whether we are fully prepared to take immediate advantage of the resulting wave of prosperity. If we are not, we shall be outstripped by our competitors. The industry is well organised, though further improvements are desirable. This is not enough; there must be the spirit of initiative and enterprise, and that cannot operate effectively if the necessary confidence in the stability of our economic structure is lacking.

While this is a matter outside the direct control of our manufacturers, the industry can render great assistance toward achieving the desired end by co-operating with the various bodies which are engaged on the task of formulating a fiscal policy to protect our home manufactures and extend our inter-Empire trade. The retained imports of chemicals into this country in 1929 was valued at £16,000,000, and there is no reason why we should not secure the greater part of this. In the same way we should make a special effort to secure the £23,000,000 worth of chemical trade with the rest of the Empire, which, in 1928, was going to the foreigner. It is a reproach to us that our present share is only 38 per cent. Important and helpful as a change in our fiscal system would undoubtedly be, we must not wait for such an uncertain factor, but endeavour forthwith by co-operative effort to explore the possibilities of many of our Empire markets, and to secure the major portion of the trade for ourselves. Only in this way can the future development of the industry be assured in face of the intensive competition which assails us from every quarter.

## Progress

By Dr. E. F. Armstrong, F.R.S.

"Of what may come hereafter  
For men that sow to reap."

The year draws to an end; we please to be retrospective; we review what has been accomplished; we ask what is the progress of a year, of a decade, of half a century—particularly in chemical industry. Perhaps we are too close to this year's results to mark the rate of advance; it is only when the definite achievement is made public, the large scale factory put into operation, or the new product launched on the market, that a notch is cut on the yard-stick of progress, though in fact the experiments, the trials, their alternating failure and success have taken possibly years to perform.

Chemical discovery, invention, achievement, seems comparable to the surge of the incoming tide on a shelving beach. Each successive wave comes a little higher; an occasional roller rushes up much further, carrying all before it; its followers do not reach so far at first, but in time, steadily persisting, they attain and pass this mark. Only the tide of chemistry never really turns, though in some directions it may appear to recede. As it comes in year by year, overcoming or sweeping aside the barriers to progress set up by ignorance, lack of education, scant appreciation, it advances always on a broader front in bigger waves with a greater surge. None can stop it—politician, peer, or populace; no one should wish to.

The tide brings its harvest on to the foreshore, shoals of fish, wrack for the land, flotsam and jetsam, or maybe the spoil of a wreck. The chemical tide is more beneficent; its spoils are more certain, more utilitarian; it also has its wrecks.

### Looking Backward

Chemical industry is fortunate to-day in being largely championed by young men who, according to Plato, are the truest philosophers, not because they necessarily philosophise well, but because they rush upon ideas with their whole soul. No wonder that it is progressing more speedily than ever before, faster probably than any other industry has ever advanced.

If we go back to the beginning of the nineteenth century, when the steam engine was the wonder of the day, the only method of travel was on the roads, created as the trackways of the ancient Britons, modified for military purposes by the Romans; the canals then furnished the sole alternative means of transport. Men could travel no faster either by land or sea than had been the custom almost from the very beginning of classical history. From the time of the birth of the railways, corresponding with the accession of Queen Victoria right up till her death at the end of the century, the iron roads of the railways held sway and the old coach routes were all but deserted.

Then came the advent of the internal combustion engine and the automobile, progressing at first slowly but now at such a pace that the railways in their turn are menaced, perhaps more gravely than is yet realised. Better, broader, faster roads are a necessity; the existing highways are entirely inadequate for the ever-growing stream of heavy vehicles, public and private cars, that beset them.

The provision of roads has brought with it many problems for the chemist, who has even been a little late in realising and grappling with them. The questions of concrete or macadam, of resistance to water penetration, of opposition to skid, all concern him. The chemistry and physics of road tar, the alternative use of asphalt, are subjects in which the year has seen progress; they are being tackled in the right co-operative spirit. Rubber roadways in the cities must be given a fair trial; success here would at the same time offer a solution of the trials of the sore beset plantation industry.

Of the automobile manufacturing industry, now as much chemical as engineering in its dependence on and utilisation of synthetic products, much has been written; this year has witnessed continuous advance.

Right in the forefront of progress is the great oil industry, whose products furnish the motive power for the twenty odd million motor cars and provide lubrication for their moving parts. A few years ago it was foretold by experts

that the oil supplies of the world were approaching exhaustion; to-day, in spite of some measure of restriction, there is a glut of oil. At first but a fraction, some 20 per cent., of the oil from the well could be refined to motor spirit; cracking processes nearly doubled the proportion so recoverable. Now this year it is said that there is the feasibility of hydrogenating the residues after cracking, so that in all some 80 per cent. of the original crude will be utilised.

Of the balance, some half is in the form of gas rich in olefines, and we hear on all sides of the recognition of its potential value as a raw material for chemical synthesis and of processes and patents for its application to this end.

The revolution in road transport will sooner or later bring with it a consideration of building, both of houses, shops and public edifices, as well as of town planning. Elsewhere much experimenting is going on in the new architecture which aims at making a building fit for the purpose for which it is to be used internally, and not a replica of some traditionally beautiful exterior. New materials are available to-day which our forbears lacked—steel, concrete, porcelain, glass, asbestos rubber, pyroxalin spray paints: all offer opportunities to the architect and the builder, of which they have so far been slow to avail themselves. Here is a field for the chemist of unlimited scope; perhaps the next decade will be one of building in which new materials supplied by the chemist substitute the present deplorable ugliness.

### Signs of Progress

During the nineteenth century, the population increased fourfold, but far more important has been the change in the general standard of living. Millions now enjoy comforts which would have been deemed unthinkable a century ago. This change is still taking place at an increasingly rapid rate; whether it is possible for the masses in this country and in Europe to attain to the standards largely persisting in the United States, whether these latter can be upheld in times of depression like the present, are some of the moot questions of the day alike for politician and economist. With the ease of modern locomotion, there comes the tendency of life to level, equalise and make uniform; it cannot be denied in this or any other country. We believe that it can only be solved through the inventive energy of the chemist making the still untapped resources of nature available for all. Vast as is the field of enterprise, there are still unknown, unworked portions where discovery is possible, and the salt of success has not lost its savour.

The urge of invention largely follows the tide of fashion, perhaps because it is easier to obtain the necessary finance for experiments in fields of inquiry which are popular and therefore easily made comprehensible to the investor. Of course, some work is going on at all times in every direction, but except in boom times rapid development is often delayed by financial considerations. The Great War was the only period in history when monetary considerations were of no importance, and consequently invention received a stimulus of the most far-reaching character. It is to this more than anything else perhaps that the jump forward in technical progress in almost every direction during the past decade is due, and its abnormal rate has been partly maintained by the unprecedented prosperity of the United States during the period. In Germany, owing to diminished prosperity, the aftermath of inflation and currency depreciation, the tide of invention has slackened in relation to its pre-war activity.

Whilst at the moment most of the apparent advance is in the fields indicated, a few years ago chemical progress was particularly associated with textiles, viscose, acetyl silk, dyes, and their application and all the side issues appertaining thereto. At another time, carbonisation questions held the field, and perhaps these will come again, for something must be done to solve the coal question with its tragic economic, political and sociological encumbrances.

When bad times come, "curtail research" was the old adage—the wise man, besides knowing all those things which the books tell us he does, knows also that then is the time to increase research. Are we doing this here in Britain? We fear not, though we are told America is.

The relation between chemical practice and theory is always interesting. To-day more than ever, now that we have catalysts in our pockets, it is possible to perform many reactions which theory either did not anticipate or immediately explain. But such happenings make it more than ever necessary to understand and establish the fundamentals of industry. It is in this direction that the various joint research associations of the D.S.I.R. and the manufacturers are doing such useful work. It is possible that this is not always appreciated at its full value by manufacturers anxious to obtain immediate returns on their investment; but, particularly because much of the work done is of a kind which cannot be carried out in a works laboratory, it is all the more valuable. Anyone with vision, acquainted with the needs of the textile industry, will

have little hesitation in appraising the work done at the Shirley Institute as of the highest value.

The chemist is content as a whole to work, to originate, to invent, often letting the fruits and results of his efforts be abused or misappropriated by others. It is a moot point whether he should not come more into the open, be heard in the councils of the nations, be listened to with respect when he warns or scolds.

We must have the courage to disapprove: acceptance is so easy. Bertrand Russell, in his *Sceptical Essays*, reminds us "that we may hereafter learn to create virtue by manipulating the ductless glands and stimulating or restraining their secretions. For the present it is easier to create a scientific habit of mind in forecasting the effects of our actions."

## The Dyestuffs Act: Ten Years—and After

By W. J. U. Woolcock, C.B.E.

(Chairman of the Dyestuffs Industry Development Committee.)

*This comprehensive and carefully reasoned survey of the dyestuffs situation was written when it seemed likely that the Government's decision to allow the Act to lapse would be final. Later, however, that decision was reversed, and the Act will continue in force for another year. Readers will bear this in mind in relation to any references to the lapse of the Act.*

I MAKE no apology for again introducing a topic which has been the subject of so much discussion and comment during the past few weeks—exceedingly critical weeks so far as the dyestuffs industry of this country is concerned. Indeed, I feel that there is now every reason why the subject should be aired on every possible occasion, having regard to the disappointing results of the efforts of those well-versed in the question, to bring home to the guardians of the nation's welfare and prosperity the importance of this question.

Notwithstanding that all the relevant facts have been reviewed in the fullest possible manner so recently, I may, perhaps, be permitted to indulge in a brief recapitulation here.

### Reasons for the Act

The events antecedent to the passing of the Dyestuffs (Import Regulation) Act will be fresh in the minds of all. It will be remembered that at the end of 1919 Mr. Justice Sankey declared that the exclusion of foreign dyestuffs under the Prohibition of Imports proclamation of February of that year was illegal, and that for a period of a little over 12 months no restrictions whatever stood in the way of importation of foreign dyestuffs, of which very large quantities were brought into this country. The dyestuffs industry of Great Britain was thereby threatened with extinction until the Dyestuffs Act became law in December 1920.

### Administration and Operation of the Act

The Act prohibits importation into the United Kingdom of all synthetic organic dyestuffs, colours, and colouring matters, and all organic intermediate products used in their manufacture, except under licence granted by the Board of Trade on the recommendation of the Dyestuffs Advisory Licensing Committee. This Committee consists of five representatives of the colour-using industries; three representatives of the dye manufacturing industries; and three independent persons of whom one is the chairman. It will be observed from this constitution that if a user considers that he has a case for importing a foreign product, it is only necessary for the consumer members of the committee to convince one of the independent members in order to get a majority. On the other hand, in order to gain their point, it is necessary for the manufacturers' representatives to win over to their side all three of the independent members.

### The Licensing Committee

In practice the Licensing Committee has granted a licence to import a foreign product on one of two grounds, namely, where (1) no British-made equivalent or adequate substitute of satisfactory quality was available, or (2) there was a satisfactory British equivalent, but at a price which in the opinion of the Committee would have placed the consumer in an unduly disadvantageous competitive position.

With the object of defining what constituted such a position the Committee, in the second year of the working of the Act, introduced a factor system for dealing with applications for licences on price grounds. At the time of the inauguration of

this system the factor fixed upon was three, and a consumer was entitled to an import licence wherever the price of the relevant British product was in excess of three times the pre-war price paid for the corresponding foreign product, or the current quoted price, whichever happened to be the higher. The factor has been steadily reduced until it now stands at 1.75. It should be pointed out that the factor price is a maximum and not a minimum price, and that some 80 per cent. by weight of the total of British-made dyestuffs are being sold at prices well below the maximum which the Committee's system would permit them to be sold at. Moreover, for some years now the British prices have compared so favourably with those of foreign competitors that requests from consumers to be allowed to import products for reasons of price have been very few.

### Progress since the Act

In judging the progress made during the period of protection, it is important to bear in mind that during the earlier years there were certain circumstances which militated against development. For example, £7,000,000 worth of foreign dyestuffs were imported into the country in the interval between the Sankey Judgment and the passing of the Dyestuffs Act. Also, very large quantities of German dyestuffs were brought into the country on Reparations account. It will readily be understood that these were a source of very considerable embarrassment to the British makers of dyestuffs. Moreover, the abnormal conditions of trade during 1921 and 1922 acted detrimentally in so far as they were not conducive to expenditure either on new plant or on research.

During the period of operation of the Act the British dyestuffs industry has progressed to such an extent that it is now producing more than 55 million pounds weight of dyestuffs annually as compared with 24 million pounds in 1922, and a little over 9 million pounds in 1913. In comparison with the pre-war output the present figure of production represents an even greater advance than the figures themselves suggest, because a very considerable proportion of the 1913 output consisted of dyestuffs made from intermediates imported from abroad. The present production, on the other hand, is made almost entirely from intermediates manufactured in our own dyestuffs factories. The proportion of the total dyestuffs consumption of this country which is now being supplied by the home industry is 93 per cent. by weight and about 80 per cent. by value, as compared with between 10 and 20 per cent. by weight in 1913.

The quality of dyestuffs now manufactured here is admitted to be at least equal to that of competing foreign products. The range is continuously expanding, and it is worth mentioning that the most marked advance in recent years has been in the most complicated and fastest series, namely, the Vat colours, for which there is a daily increasing demand.

There still remain gaps in the British range, however, and these are important not only because of the individual numbers thus lacking to consumers, but even more because



Mr. J. Arthur Reavell, President, Institution of Chemical Engineers.



Dr. G. C. Clayton, President, Institute of Chemistry.



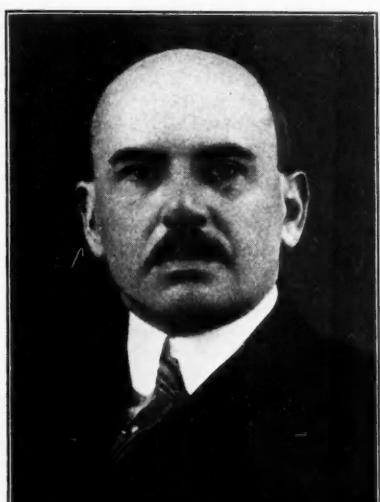
Professor J. F. Thorpe, F.R.S., President, Chemical Society.



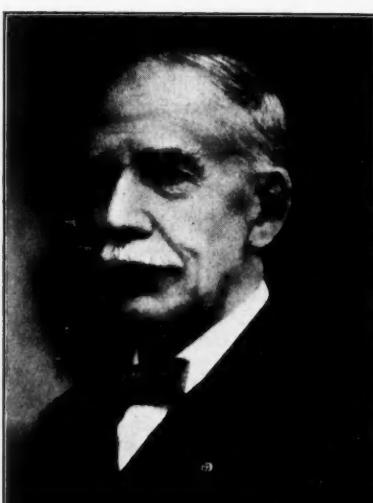
Mr. W. J. U. Woolcock, Chairman, Dyestuffs Industry Development Committee.



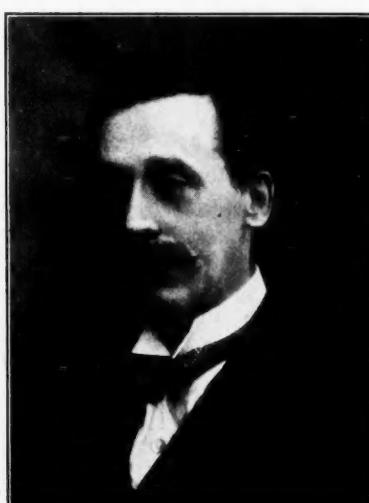
Mr. J. Davidson Pratt, General Manager, A.B.C.M.



Mr. A. F. Butler, Chairman, British Chemical and Dyestuffs Traders' Association.



Dr. J. T. Dunn, President, Society of Public Analysts.



Dr. G. T. Morgan, Superintendent, Chemical Research Laboratory, Teddington.



Mr. F. Scholefield, President, British Association of Chemists.

of the fact that they include some categories the manufacture of which requires special technique. In some of these research work is now well advanced. In others the manufacturing stage has already been arrived at.

Too much stress cannot be laid upon the fact that although the British industry has had to comply with the Users' demands for existing foreign types, a considerable amount of valuable original work has been done, resulting in some entirely new discoveries of great commercial value.

The constant downward trend of prices bears very eloquent testimony to the strenuous efforts of the industry. During the year prior to the passing of the Act the average price of dyestuffs in this country was 4s. 4d. per pound. From that time prices have continuously declined, and at the end of 1928 the weighted average price of all British dyestuffs had dropped to 1s. 6½d. per pound, or slightly more than one-and-a-half times the pre-war average price. Since 1928 further reductions have taken place. This achievement in lowering prices is largely due to the stable conditions which the Act has secured for the industry, and the placing of bulk orders which has permitted production on a large scale and utilisation of plant under the most economic conditions. There is reason to believe that the operations of British dye-makers have constituted a powerful instrument in reducing foreign dyestuffs prices.

#### Threatened Lapse of the Act

In their final report to the Government the Dyestuffs Industry Development Committee, appointed by the Board of Trade to advise them with respect to the efficient and economical development of the dye-making industry, have included the following statement amongst their conclusions:—

"Consequently it is to the interest of all parties concerned, that is the Government, the Users and the Dyestuff manufacturers, to continue to consider the problem in the same spirit of co-operation that has marked the period of the operation of the Dyestuffs Act, and together to agree, if possible, first whether any further assistance to the industry is necessary, and, if so, as to the form which such assistance should take."

Agreement concerning the future of the Act between the parties named not having been possible, the dye-makers, in urging their case for a continuation of the Act, have made a definite offer that in the event of the Act being renewed prohibition of import shall only apply where a British maker is prepared to supply an equivalent product at an equal price to that quoted for the foreign product. This offer, which is subject to safeguards against unfair prices being quoted with the object of obstructing the development of the industry in this country, removes all traces of the Users' objection on the score of price. They have already admitted that so far as quality is concerned British products are well up to the world standard.

The offer has not been accepted. On the other hand, the chairman of the Colour Users' Association has made a suggestion of extending the Dyestuffs Act for a few years, but at the same time restricting its protection substantially to those dyestuffs which will be in manufacture in this country on January 14, 1931, and available at world competitive prices. This proposal, as formulated, appears to contain many grave disadvantages from the dye-makers' point of view, and to introduce serious difficulties in the way of administration. In any case it is not a proposal put forward by the official body of dyestuff consumers—the Colour Users' Association.

In spite of the cogent arguments which were put forward by those who were alive to the importance of ensuring the complete and firm establishment of the dyestuffs industry in this country, the Government's decision to allow the Act to lapse on its expiry on January 14, 1931, was confirmed after a debate lasting more than four hours in the House of Commons on December 4.\*

Let us examine the three main arguments advanced by the supporters of the Government's decision:—

#### (1) That the Act was passed for 10 years and no longer.

The supporters of an Amendment to continue the Act for a further period pointed out that several circumstances which could not have been foreseen by the framers of the Act at the time when it was passed have operated against the industry reaping the full benefits intended. They urged that owing to these circumstances they had not, in fact, enjoyed an

effective 10 years of production and that they were entitled to ask that the question should be considered in the light of world conditions as they are to-day and not as they were 10 years ago. In any case, there is no such thing as finality in the industry of dyestuffs manufacture, and the framers of the Act could not possibly have foreseen the extent of the new ground which the industry would have to cover in the 10 years, over and above the task of catching up with the foreign industry, as it already stood at the beginning of 1921.

#### (2) That an efficient dyestuffs industry has now been firmly established in Great Britain.

It is true that the British industry is now so far established as to be able to supply over 90 per cent. by weight of the total dyestuffs consumption of the country, and to sell its products at prices which compare favourably with those paid by consumers in other parts of the world. It is to be remembered, however, that the proportion calculated by value represents only 80 per cent. of the total consumption. The imports of dyestuffs still amount to upwards of £1,000,000 annually.

The advocates of the extension of the Act maintain that the industry is not sufficiently firmly established in this country to enable it to meet, unaided, international competition, and especially such as may be expected from the powerful German organisation which has the advantage of more than 50 years' experience and practice. Moreover, the Government's supporters assume that because the British industry has arrived at a stage when it would provide the above large proportion of the requirements of the consuming trades at prices competitive with world prices *under existing conditions*, it is therefore firmly established. They appear to have completely overlooked the fundamentally important fact that this favourable price position is only possible if the British manufacturers are assured of the bulk of the business, and thus permitted to manufacture their products in such quantities as will enable them to run their plants economically and efficiently. Any change in the existing conditions in the direction of increase in the proportion of foreign dyestuffs will seriously affect the economic working of the domestic industry.

#### (3) The Act is a burden to the textile industries of the country and adversely affects their trade, especially in foreign markets.

This argument appears to be the one which weighs the most with the Government's supporters. It is evident that they have attached the greatest importance to representations made by various organisations in the textile trades, especially the Lancashire cotton industry. It is an anomalous fact that these representations have come chiefly from sections of the textile industries which do not themselves use dyestuffs and which are therefore ill-qualified to express views as to the effect of the dyestuffs question upon their trade.

The actual users of dyestuffs are divided in opinion, and moreover, the reasons of those who have opposed the renewal of the Act are based upon conditions of the past and not upon those which obtain to-day.

Abundant evidence has been produced by the supporters of an extension of the Act, that the cost of dyestuffs constitutes but a very small fraction of the total cost of production of textile goods, and that the contention which has been made that the loss of trade in coloured cotton goods in foreign markets is due to the operation of the Act, is quite untenable.

It is a significant fact that in the very comprehensive Report (1930) of the exhaustive investigations of the Committee on the Cotton Industry into the cases of the loss of Lancashire's trade, not a single suggestion is made that the question of dyestuffs is in any degree concerned.

#### Attitude of Opponents

The speeches of the opponents of a continuation of import restrictions showed quite clearly that, in spite of the great prominence which has been given to this aspect of the matter they neither appreciate nor understand the profound bearing of the firm establishment of an efficient dyestuffs industry upon national safety and the general welfare of the people. As regards the former they take up the attitude that the question of safeguarding the country in the event of hostilities is entirely one for the Department of the Government directly concerned to look after and to provide for out of public funds. They completely ignore the fact that in the first place, no government can forecast with any degree of accuracy, what instruments of war might be called into commission by a belligerent power. *Ad hoc* research alone would be futile to cope with this question. They ignore the known fact that

\* This decision was later reversed.

adequate dyestuffs plants, running efficiently in times of peace, are those which, in times of war, can almost instantly and automatically be switched over to the manufacture of munitions and other instruments of war as we know them at the present time. The general welfare of the nation is by far the most appealing aspect of the case. The truth about its close connection with the fostering of organic chemical research, which is mainly rendered possible by the existence of an efficient and powerful dyestuffs industry, has been widely disseminated during recent years. It is well-nigh incredible that the Government's supporters should still be in such deplorable ignorance of the facts as to suggest, as they have done, that (1) the importance of a dyestuffs industry to the conduct of efficient and adequate organic chemical research has been grossly overstated, and (2) any necessary research can, and should be, provided for by such institutions as the Privy Council Department of Scientific and Industrial Research. It is only too evident that those who make such suggestions lack even the most elementary knowledge of the matter. That they have not considered the advisability of consulting the opinion of those who are entitled to be regarded as well-informed on these questions is amazing in the extreme.

In the minds of those who have pressed for a further period of protection for the insufficiently established dyestuffs industry there is present the very real fear that their old-established foreign competitors will not hesitate to have recourse to dumping of their products to this country, in order to handicap our industry and prevent it from developing to the stage when it could meet them on level ground. The supporters of the Government's decision refuse to believe in the possibility of dumping taking place. At the same time they suggest that if, after the Act has lapsed, the Government are satisfied that foreign dyestuffs are being dumped here,

they will be prepared to take remedial measures. But it is difficult to see how this could be done before the industry had been severely hit. In the space of a few months, Germany, for example, could send into this country sufficient quantities of their products to cause grave dislocation of British dyestuffs manufacture. Much damage would be done before any remedial measures could be applied.

Important as is the establishment of a dyestuffs industry to the immediate consumers of dyestuffs, it will, I think, be admitted by all who have carefully studied the subject, that by far the most important aspect of the industry is its bearing upon the state of organic chemistry and the prosecution of organic chemical research, and their combined influence upon the nation's safety, welfare and advancement.

#### Postscript

So much was written while the fight was still on, and now it is over. The Dyestuffs Act is to be continued for one year. That the dyemakers have won is important, but it is not of the greatest importance. What really matters is to follow. Will the colour users, who admittedly have paid a price for the establishment of the British industry, unanimously and wholeheartedly throw in their lot with the British dyemakers and determine once and for all that British textiles shall be dyed with British dyes? Then their great industry, in value one hundred times as large as the dyestuffs industry, will march forward hand in hand with that industry. And workers in the textile industries which do not actually use dyestuffs will be thankful that the specious arguments put forth on their behalf were demonstrably false. Once more, provided the real factors in the depression in Lancashire trade are dealt with, there is a chance to stop the rot in the unemployment figures in that county.

## The Nitrogen Industry in 1930

By E. B. Maxted, <sup>D.Sc., Ph.D., F.I.C.</sup>

*Dr. Maxted, in his review of technical developments in the nitrogen industry, indicates the chief advances, and claims that the volume of work on nitrogen compounds is at least as great as in the preceding year.*

ACCORDING to the annual report of the British Sulphate of Ammonia Federation, the world nitrogen consumption for the fertiliser year 1929/30, which is of the order of 2½ million tons, has increased by only 4½ per cent., compared with increases of 14 per cent. and 25 per cent. respectively, for the fertiliser years 1928/29 and 1927/28. This gradual falling off in the magnitude of the annual increase may obviously be interpreted as indicative of an approach to the saturation of the world nitrogen market; but its real roots would appear rather to lie—as has also been expressed in the above report—in the economic position of farmers who, in view of the fall of the prices of agricultural produce, have curtailed their expenditure generally, including the purchasing of fertilisers.

The opinion that this fall is, firstly, merely a psychological time-lag in combating less favourable conditions by a greater, rather than lesser, employment of fertilisers and, secondly, one which can be corrected by educational propaganda, is strengthened by the coming into operation—in spite of this apparent present overproduction—of a certain number of new plants. Thus, it is reported that Imperial Chemical Industries, Ltd., are to erect a South African plant at Modderfontein, while a new Mont-Cenis plant, having a capacity of 10,000 tons of fixed nitrogen per annum, is to be operated in California. Other nitrogen projects include a new Fausser plant at Tarnow, Poland, with a production of 20,000 tons of ammonia per annum and a larger factory in Manchuria (1).

At the same time, various measures have, in view of the general world conditions, been adopted to limit these extensions. The German Stickstoff Syndikat, which controls 98 per cent. of the total fixed nitrogen produced in that country—amounting to a figure of the order of 1 million tons per annum—has provided for a gradual increase during the next seven years to 1·6 million tons; and the world price of nitrogen has been fixed by international agreements.

It is interesting to note that J. B. Deakin, in his presidential address to the Coke Oven Managers Association (2), states

that by-product ammonium sulphate has almost ceased to be remunerative; but this is, of course, dependent on the relative values which are assigned to the various products obtained in the coking of coal. In Chile the production of nitrate has remained approximately the same; but exports have considerably increased (3), and economies have been introduced by the closing down of certain officinas where the cost of production is abnormally high.

The following patents and papers published during the year may be noted:

#### Synthesis of Ammonia

In the manufacture of gas for the synthesis of ammonia, the modern trend is probably towards the low-temperature separation of coke-oven gas, in place of the manufacture of hydrogen by the interaction of water-gas or producer-gas with steam. The case for electrolytic hydrogen from off-peak power has been discussed by F. P. McMichael (4). This source of power suffers of course from the disadvantage of not being continuously available. Reference may be made to an interesting method of combining methyl alcohol manufacture with the preparation of the gases required for the synthesis of ammonia. Water-gas is produced in the ordinary way, and from this, the carbon monoxide is abstracted by a methyl alcohol synthesis. The residual gas is employed for the manufacture of ammonia.

The engineering side of the synthesis, with special reference to types of compressors actually used in Germany, has been dealt with by E. Blau (5). Other mechanical details include the proposal of Ernst and Young (6) to use liquid ammonia itself for the lubrication for the circulating compressors. It has long been known that other liquefied gases act as efficient lubricants; for instance, in certain types of cooling plant, employing liquid sulphur dioxide, no other form of lubricant is employed. A further point concerns the avoidance of decomposition

(3) THE CHEMICAL AGE, Vol. XXII, p. 45.

(4) *Chem. and Met. Eng.*, 1930, **37**, 484.

(5) *Chemiker Ztg.*, 1930, **54**, 2, 20, 66.

(6) U.S. Pat., 1,739,957.

(1) *Chemistry and Industry*, 1930, **49**, 381.  
(2) THE CHEMICAL AGE, Vol. XXII, p. 404.

of ammonia on hot metal parts by lining these either with silicon itself or with cements containing free silicon (7). Water glass may be used as a binding material. Finally, several furnaces for carrying out the synthesis have been described. The Union Chimique Belge (8) have protected a furnace of the type in which a part only of the incoming gases pass through an internal heat exchanger, while a part is introduced directly into the catalyst space. By this means an effective control of temperature is obtained. O. Piette describes a special design of electrically heated furnace (9); and a laboratory furnace with heating elements which are adjustable without releasing the pressure has been designed by Morgan and Tongue (10).

The heating of the circuit gases by exchange partly against compressed nitrogen heated by means of a lead bath is dealt with in a patent granted to the Gasverarbeitungsgesellschaft (11). In connection with the heating of furnaces generally by compressed nitrogen in this way, it may be noted that gases, at pressures such as are used for the synthesis of ammonia, have a heat value per unit of volume sufficiently high to render this method extremely effective and convenient. A somewhat similar division of the cooling of the circuit gases (for separating the ammonia formed), in that the first stages are carried out by indirect water cooling and subsequent stages by indirect contact with liquid ammonia, which is evaporated at a low pressure, has been proposed by the Interessen-Gemeinschaft (12).

No radically new proposals for ammonia catalysts have appeared during the year. Catalysts with an iron base seem to be firmly established; and the many possible variations of raw materials, promoters and methods of preparation reviewed in previous reports seem, for the time being, to have become exhausted.

#### Ammonium Salts

In the introduction of the present review, reference has been made to the less favourable economic position of by-product sulphate. An interesting analysis of the economics of by-product sulphate manufacture by individual gasworks has been contributed by P. Parrish (13). The factors which determine the selling of the ammoniacal liquor as such or its conversion into sulphate are discussed in detail, for which reference should be made to the original paper. W. Adam and the Gas, Light and Coke Co. (14) describe the preparation of ammonium sulphate in elongated crystals which do not cake, by adding to the hot solution small quantities of solutions of the salts of iron, chromium, aluminium or certain other metals, in the presence of free acid. D. Tyrer and Imperial Chemical Industries, Ltd. (15), have protected plant for the removal of ammonia from coke oven gas; and Wasiblewski (16) and others have described a semi-technical plant for the continuous preparation of ammonium sulphate from gypsum. In this, the operation is carried out at a pressure of 2-3 atmospheres and the temperature is gradually increased up to 120°. This formation of ammonium sulphate from ammonia, carbon dioxide and calcium sulphate is, of course, of great interest as an alternative to the direct method, particularly in connection with synthetic ammonia, although it is obviously not confined to ammonia from this source. Finally, the formation of sulphate by oxidising with air ammonium sulphite dissolved in alcohol is dealt with in a patent of the I.G. (17).

Passing to ammonium chloride, an analogous process to that described above for the sulphate, namely, the addition of small quantities of chlorides of iron, nickel, cobalt, manganese, etc., with which ammonium chloride can form mixed crystals or double salts, is stated (18) to facilitate the production of

- (7) Brit. Pat. 325,557.
- (8) Brit. Pat. 329,551; *THE CHEMICAL AGE*, XXII, 86.
- (9) Brit. Pat. 332,532; *THE CHEMICAL AGE*, Vol. XXIII, p. 291.
- (10) Brit. Pat. 325,678; *THE CHEMICAL AGE*, Vol. XXII, p. 400.
- (11) Brit. Pat. 307,027.
- (12) Brit. Pat. 329,079; *THE CHEMICAL AGE*, Vol. XXIII, p. 55.
- (13) *THE CHEMICAL AGE*, Vol. XXII, pp. 170 and 608.
- (14) Brit. Pats. 330,945 and 330,947; *THE CHEMICAL AGE*, Vol. XXIII, p. 170.
- (15) Brit. Pat. 322,049; *THE CHEMICAL AGE*, Vol. XXII, p. 83.
- (16) *Przemysl Chemiczny*, 1930, **14**, 150.
- (17) Ger. Pat. 453,686.
- (18) Imperial Chemical Industries, Ltd., and C. W. Bunn, (Brit. Pat. 326,642; *THE CHEMICAL AGE*, Vol. XXII, p. 468.

uniform small crystals of ammonium chloride. A method of manufacturing solid ammonium chloride, without evaporation of a solution, is due to J. I. Brönn and the Concordia Bergbau A.-G. (19). Gaseous hydrochloric acid and moist ammonia are brought together at a temperature above 120°, when solid ammonium chloride separates from the steam.

Of other ammonium salts the carbamate is of special interest in view of the availability at certain synthetic ammonia factories of large quantities of waste carbon dioxide; and attention may be called to a specification of the I.G. in which the preparation of this salt in solvents such as methyl alcohol, in which ammonium carbamate is substantially insoluble (20). A further solvent of this type is calcium chloride solution previously saturated with ammonia. Ammonium carbamate is precipitated directly. Certain non-aqueous solvents, such as liquid ammonia or methyl alcohol, may also be used for the manufacture of other ammonium salts by double decomposition (21). Thus from sodium or calcium chloride and ammonium carbamate ammonium chloride is obtained. The use of liquid ammonia, either with or without ammonium nitrate, to vary the solubility of salts with the object of separation, has also been dealt with (22); indeed, the changed conditions of solubility which arise by the use of liquid ammonia in place of water are only now beginning to be utilised technically.

A method of precipitating solid ammonium formate without the necessity for evaporation has been described by the Lonza Elektrizitätswerke (23), according to which dry calcium formate is added to ammonium formate solution at -10°, followed by carbon dioxide and ammonia, or ammonium carbonate, the temperature being raised to 100°. Calcium carbonate is precipitated and removed, whereupon the solution is once more cooled to -10°, when ammonium formate separates out.

Various specifications have been published dealing with the manufacture of mixed ammonium salts, mainly for use as fertilisers. Thus, the I.G. (24) employ double decomposition in liquid ammonia in a manner somewhat similar to that already noted, but with the production of a mixed product. From sodium chloride and ammonium nitrate, ammonium chloride and sodium nitrate, together with some unchanged salts are obtained; potassium phosphate and ammonium nitrate give ammonium phosphate and potassium nitrate. A further method preparing ammonium phosphate suitable for fertilisers has been described by the Société Chimique de la Grande Paroisse (25). In this method sodium phosphate is treated with ammonia, carbon dioxide, and water under conditions for which the original specification should be consulted.

The production of a mixed fertiliser from diammonium phosphate and ammonium nitrate forms the subject of a patent specification due to the I.G., in which is included plant suitable for the production of the mixed salt in the form of small globules, by spraying. Another patent of the same firm (26) covers a method for the manufacture of mixed ammonium and potassium nitrate by treating potassium carbonate with nitric acid and ammonia under special conditions.

#### Nitric Acid and Nitrates

Dealing first of all with the formation of nitric acid by the oxidation of ammonia, considerable interest is attached—by reason of their success in other oxidation reactions, particularly for the oxidation of sulphur dioxide—to catalysts containing vanadium. The Selden Company and A. O. Jaeger (27) give examples of a number of catalysts having a zeolite base and containing vanadates, tungstates or chromates. The activities of such bodies relative to platinum, from the standpoint of capital cost, optimum rate of passage and yield of nitric oxide, are of great technical importance. Improvements in the activity of platinum itself are claimed if the

- (19) *THE CHEMICAL AGE*, Vol. XXIII, p. 332.
- (20) Brit. Pat. 329,737.
- (21) Brit. Pat. 332,584; *THE CHEMICAL AGE*, Vol. XXIII, p. 291.
- (22) Brit. Pat. 331,236; I.G., *THE CHEMICAL AGE*, Vol. XXIII, p. 192.
- (23) Brit. Pat. 323,113; *THE CHEMICAL AGE*, Vol. XXII, p. 182.
- (24) Brit. Pat. 329,641; *THE CHEMICAL AGE*, Vol. XXIII, p. 86.
- (25) Brit. Pat. 332,864; *THE CHEMICAL AGE*, Vol. XXIII, p. 312.
- (26) Brit. Pat. 329,883; *THE CHEMICAL AGE*, Vol. XXIII, p. 105.
- (27) Brit. Pat. 313,153.

surface is covered with rhodium (28). The combined catalyst may also be made by using platinum as the covering metal. For the manufacture of pure oxides of nitrogen, for transformation into highly concentrated nitric acid, oxygen, where available as a by-product, possesses certain advantages over air, since the product is not diluted with nitrogen. In order to prevent explosion, a burner of special type must be used; and a further design for this has been published by J. Y. Yee (29). The burner itself is of aluminium and water-cooled. It should be noted that platinum-rhodium alloys and a burner for utilising oxygen, in each case based on the work of investigators other than those now cited, were mentioned in last year's report.

Another suggestion for obtaining the oxides of nitrogen in a concentrated form, even when air is used as the oxidising medium, is due to L. Bergfeld (30). The gases from the oxidation chamber are absorbed by means of solutions of compounds of chromium or manganese; on heating the nitrogen oxides are evolved in a pure condition.

An interesting preparation of nitrous oxide by direct oxidation of ammonia has been described (31). Air or oxygen, together with sufficient ammonia for this oxide, is passed over platinum at a relatively low temperature (200—400°). The product is stated to be free from higher oxide, and is separated by cooling or by adsorption in silica gel. In connection with the formation of nitrogen oxides, it may be noticed that an explosion apparatus for the production of these bodies from air has been designed by H. A. Humphrey (32).

The absorption of oxides of nitrogen form the subject of several patents published during the year. Thus a modification in tower design has been protected by the Hercules Powder Co. (33); and, according to a proposal of the I.G., the nitrous gases are brought, without being cooled, into contact with basic absorbents, nitrates and nitrites being produced directly in a solid form. Improvements in the concentration of nitric acid by enrichment with nitrogen peroxide in the presence of oxygen, continue to receive some attention; and special variations in temperature and other conditions conducive to better working are described in Brit. Pat. 321,278 (34).

For the purification of nitric acid from traces of sulphuric acid, the I.G. (35) add salts of metals the nitrates of which are soluble but the sulphates insoluble, when a precipitate of the metallic sulphate is produced. Salts or oxides of magnesium, aluminium, iron, zinc, manganese or nickel are stated to be suitable.

The conversion of potassium chloride to the nitrate forms the subject of several patent specifications published during the year. Thus, double decomposition between the above chloride and calcium nitrate (36) is claimed to be more complete if it is carried out in the presence of ammonia, the solution being preferably cooled to a temperature which may be as low as —30°. After separation of the potassium nitrate, a further addition of ammonia is made to precipitate  $\text{CaCl}_2 \cdot 6\text{NH}_3$ , in order to render possible a further working up of the liquor to obtain more potassium nitrate. P. Höfer and the Kali-Forschungs-Anstalt (37) mix hot solutions of potassium chloride and a metallic nitrate which is easily hydrolysed (e.g., aluminium). Potassium nitrate separates out on cooling, following which the mother liquor is evaporated, with evolution of hydrochloric acid until most of the chlorine has been expelled. Nitric acid and water are then added until the original concentration of the metallic nitrate is re-attained, when the cycle of operations may be repeated. A further method of treating potassium chloride, with nitrous gases in the presence of alumina, is contained in Brit. Pat. 327,909 (38).

In the preparation of potassium nitrate from nitric

(28) I.G. Brit. Pat. 331,728; THE CHEMICAL AGE, Vol. XXIII, p. 234. See also Brit. Pat. 334,466; THE CHEMICAL AGE, Vol. XXIII, p. 413.

(29) *J. Ind. and Eng. Chem.*, 1929, **21**, 1024.

(30) Brit. Pat. 324,220; THE CHEMICAL AGE, Vol. XXII, p. 302.

(31) I.G. Brit. Pat. 325,475; THE CHEMICAL AGE, Vol. XXII, p. 398.

(32) Brit. Pat. 330,273; THE CHEMICAL AGE, Vol. XXIII, p. 124.

(33) Brit. Pat. 329,427; THE CHEMICAL AGE, Vol. XXIII, p. 86.

(34) THE CHEMICAL AGE, Vol. XXII, p. 57.

(35) Brit. Pat. 328,156; THE CHEMICAL AGE, Vol. XXII, p. 612.

(36) Brit. Pat. 328,272; THE CHEMICAL AGE, Vol. XXII, p. 612.

(37) Brit. Pat. 332,359; THE CHEMICAL AGE, Vol. XXIII, p. 259.

(38) O. Kawelitz and The Kali-Forschungs-Anstalt, THE CHEMICAL AGE, Vol. XXII, p. 581.

acid and potassium chloride, nitrosyl chloride is evolved. This, according to two further patents of the Kali-Forschungs-Anstalt, is absorbed either by hot quicklime—by which means calcium chloride is formed and oxides of nitrogen recovered—or by passage over silica gel (39).

Another patent dealing with nitrates, but of a completely different type, is due to G. H. Gleason (40), according to which crude sodium nitrate is raised to a temperature just above its melting point. Most of the impurities remain solid and do not dissolve in the molten nitrate, which may be sprayed in the form of globules of suitable size.

#### Other Nitrogen Compounds

The principal bodies which fall in this group are hydrocyanic acid, calcium cyanamide and urea. The Imperial Chemical Industries, Ltd. and others describe two methods of preparation of hydrocyanic acid from hydrocarbons. According to one process, methane or ethane is passed, with more than one molecule of ammonia for each carbon atom present, through unpacked tubes of refractory material, heated to about 1,150° C. (41). In place of ammonia, nitrogen may be used (42), the mixture of hydrocarbon and nitrogen, preferably originally containing a little free oxygen, being heated to a temperature above 800° C. and subjected to the action of an electric arc. A process, due to the I.G., for the preparation of nitriles may also be noted. A mixture of acetylene and amines (43) is passed over heated compounds of zinc, vanadium, aluminium, or certain other elements (see also Brit. Pat. 259,276; THE CHEMICAL AGE, Vol. 19, p. 369).

Various special methods of obtaining calcium cyanamide from calcium carbonate and ammonia have been described. Thus, J. Raitznye (44) passes ammonia over calcium carbonate, containing a small percentage of iron, at 600—800°; further, the I.G. (45) maintain the partial pressure of carbon dioxide, and of any water vapour present, at as low a value as possible during the reaction with nitrogen. For other suggested conditions, reference is made to Brit. Pat. 326,117 (46). A process and plant for the continuous production of calcium cyanamide from calcium carbide and nitrogen in a shaft furnace is due to H. Wittek (47). Calcium carbide is charged into the centre portion of the furnace, and the cyanamide withdrawn continuously from the end.

Finally, mixtures of urea and other bodies as fertilisers are dealt with by the I.G. in several specifications (48); and Kruse, Gaddy and Clark (49) have contributed an interesting description of a direct process for the production of urea from liquid ammonia and liquid carbon dioxide by treatment in a lead-lined autoclave at 150° C.

It will be seen from the above summary that the volume of work on nitrogen compounds is at least as great as in the preceding year. Its nature has, particularly with the stabilisation of the synthetic ammonia industry, necessarily undergone modifications compared with the fields of work which were of topical interest, for instance, ten years ago. Little or nothing new is heard of the alternative methods of nitrogen fixation which were then being investigated or actually in operation—the Bucher process for the manufacture of cyanides, the production of nitrides of aluminium and other elements by high-temperature methods, and the arc process for oxides of nitrogen. The trend in industrial processes, not only in the nitrogen industry but also generally, is undoubtedly away from high-temperature reactions, wherever such a course is possible, in most cases on account of the excessive wear and tear of plant. The large-scale application of high pressures, on the other hand, is proving unexpectedly easy, economical and effective; and, as has been outlined above, attention is now being paid more and more to the possibility of carrying out double decompositions and other reactions in solvents other than water. This last trend is one which may be watched with considerable interest.

(39) Brit. Pat. 327,047; THE CHEMICAL AGE, Vol. XXII, p. 519.

(40) Brit. Pat. 315,262.

(41) Brit. Pat. 335,585; THE CHEMICAL AGE, Vol. XXIII, p. 511.

(42) Brit. Pat. 325,860; THE CHEMICAL AGE, Vol. XXII, p. 422.

(43) Brit. Pat. 332,258; THE CHEMICAL AGE, Vol. XXIII, p. 258.

(44) Fr. Pat. 628,303.

(45) Brit. Pat. 328,743; THE CHEMICAL AGE, Vol. XXIII, p. 32.

(46) THE CHEMICAL AGE, Vol. XXII, p. 442.

(47) Ger. Pat. 458,029; *Brit. Chem. Abstracts*, B, 1930, 324.

(48) Brit. Pat. 332,932, 332,945 and 332,948; THE CHEMICAL AGE, Vol. XXIII, p. 313.

(49) *J. Ind. and Eng. Chem.*, 1930, **22**, 289.

## Colour Users and the Dyestuffs Situation

### Review of an Anxious Year

*The attitude of the colour users on the Dyestuffs situation is, of course, one of the main factors, and in the following article the position is discussed, especially from the colour users' point of view.*

THE past year has been a time of great activity and critical importance for the Colour Users' Association. Consideration of the attitude to be adopted by the Association in relation to the expiration of the Dyestuffs Act and the question of the importation of foreign dyestuffs has to a large extent occupied the time and attention of the Council throughout the last twelve months.

#### The Council's Memorandum

A memorandum was prepared by the Council on the operation of the Dyestuffs Act and its effect on the colour-using industries. This memorandum has since been published, and gives in detail the colour users' point of view, together with some valuable data in the way of statistics and tables. The conclusions of the Association as set out in the memorandum are as follows:—

1. In the opinion of the Colour Users' Association, the dyemaking industry of Great Britain, under the protection of the Dyestuffs Act, has made remarkable progress, and may now be termed a virile industry. It has also to be borne in mind that the two principal manufacturing concerns which supply the major portion of the country's needs are in the strong position of being allied to combinations of great financial resources and of international renown. The Dyestuffs Act, therefore, has achieved its purpose in assisting to establish the industry in Great Britain.

2. The colour users of Great Britain have loyally honoured their pledge to assist in the establishment and development of the dye-making industry: (a) by acquiescing in high prices for dyestuffs, prices very much in excess of world prices and of the general price level of commodities; (b) by co-operation with the makers in technical matters; and (c) by assisting the Licensing Committee to render the operation of the Dyestuffs Act practicable.

3. The burden of establishing the industry has not been equitably shared. In the early stages, when the makers were called upon to lower their prices, they did so, it is understood, at considerable sacrifice. At that period the Government had an opportunity of contributing their quota toward the establishment, instead of which colour users were called upon to bear still higher burdens in the price of Reparation dyestuffs sold at rates much in excess of the general world levels, and which resulted in considerable profits to H.M. Treasury. The Colour Users' Association have on several occasions drawn the attention of the Government to this unsatisfactory state of affairs.

4. The dyemaking industry of Great Britain, whilst it is well established, does not yet provide for the full need of the colour using industries, and users are dependent upon foreign suppliers for special colours and novelties. It would be a serious blow to British users were the flow of these products to be impaired in any way.

5. The question may arise as to whether British production of dyestuffs would be imperilled after the expiry of the Act on account of a definite attack by foreign producers to regain the British market, but in the considered opinion of the Colour Users' Association such a contingency is remote, because of the importance and strength of the leading British manufacturers, nor is the serious position of 1914 likely to be repeated because many new sources of supply have been established throughout the world.

6. The Government pledge to assist in establishing the dyestuffs industry in Great Britain because of national security is an important aspect, and cannot be overlooked. Should any further assistance be necessary, surely it is a matter for which national provision should be made, and the cost should no longer fall upon the colour using industries.

At the annual meeting of the Association on July 15, 1930, the Chairman (Sir Henry Sutcliffe Smith) put forward, independently, the suggestion that if it was agreed between the interested parties that a further limited period of protection was necessary, this protection should be afforded only to those dyestuffs of which the British makers have definitely

established the manufacture in this country, down to the end of the existing Act. This list of colours to be scheduled as the protected or safeguarded list, and to include only those colours which, by agreement with the users, it has been established, are definitely equivalent to the best products made abroad, and that all other dyestuffs, both new and improvements on existing types, should be allowed free entry.

#### British Colour Council

Reference was made in the last review to the interest taken by the Association in the formation of the British Colour Council. This body has been definitely established as a limited company. Several prominent members of the Association are connected with the British Colour Council, and it is hoped that this close co-operation will result in mutual benefit to the members of both bodies.

As reported last year the Association was successful in obtaining the exemption of oxalic acid from duty under the Safeguarding of Industries Act to the end of December, 1930, and this exemption has again been renewed for a further period expiring on December 31, 1931. The procedure adopted with regard to this product and similar cases seems to call for revision. There seems no reason why a product not made in this country should be included in the list of safeguarded articles. The effect of this procedure is that the onus of obtaining exemption is placed upon the consumer, whereas it would surely be more equitable to delete the product from the schedule and leave it open for any prospective British maker to apply for its inclusion if and when production was established in this country.

#### Foreign Chemical Prices

The Association has continued to issue to its members a quarterly bulletin of chemical prices ruling abroad in comparison with those obtaining in this country. This Bulletin has been enlarged, and is of considerable value to members. In this connection, it should be noted that the Association has been responsible for a great deal of valuable work in the preparation of statistics useful to the industry—without which information it would be impossible to assess the progress of the dyestuffs industry or to ascertain its position in relation to its foreign competitors. The preparation of the colour users' case for the Report on the Dyestuffs Act issued by the Dyestuffs Industry Development Committee, and also the Association's own memorandum on the Act, entailed an enormous amount of labour on the part of the committees concerned, involving detailed consideration of the whole range and production of British dyestuffs, the collation of statistics and data from every available source, and comparison of the dyestuffs position in various countries. The value of information of this kind can hardly be over-estimated.

#### Administration of the Act

It may, perhaps, be of interest at this juncture to review briefly the work of the Association since the Dyestuffs (Import Regulation) Act came into force in January, 1921. There can be no denying the fact that the Association has been of considerable assistance to colour users during this period. Particularly is this true with regard to the operation of the licensing regulations. It was through the instrumentality of the Association that the Factor Ratio was established as a means of dealing with applications to import dyestuffs on price grounds. In September, 1922, a factor of three times pre-war level was adopted as a basis, and since then the factor has been reduced by progressive stages to the present one of 1.75. This result has been achieved largely through the initiative of the Association, which has taken an energetic part in the negotiations with the makers, and has undoubtedly been of inestimable benefit to colour users, the reduction of the factor representing very considerable savings in colour costs.

The work done by the Association's representatives on the Dyestuffs Advisory Licensing Committee and the Dyestuffs Industry Development Committee has been of great import-

ance, and through these Committees the members have been able to put their views before the Government.

No review of the work of the Association would be complete without reference to the services rendered by the Joint Technical Committee, which consists of representatives of makers and users. This Committee has done excellent work in the preparation of graded lists of colours, which have been of great assistance in enabling the Licensing Committee to deal with applications in a systematic and regular manner.

When the Safeguarding of Industries Act was placed on the Statute Book in 1921, valuable work was done by the Association's Vigilance Committee in negotiations with the Government by means of which the position was clarified and con-

cessions obtained by the deletion of certain chemicals from the lists—thereby saving colour users many thousands of pounds.

It is hardly necessary to refer to the assistance rendered by the Association to the Government in the requisitioning of dyestuffs from Germany under the Reparation Treaty, and the Association remained in close touch with the Government until the expiry of the Reparation arrangements.

This brief summary of the Association's achievements, which does not by any means include all the activities of the Association, is ample proof of the success of united action in a common interest, and is indicative of the abundant scope for the energies of the Association in safeguarding the interests of colour users.

## Recent Progress in the Gas Industry

By Sir D. Milne-Watson, LL.D., D.L.

(Governor of the Gas Light and Coke Co.)

*Sir D. Milne-Watson summarises, in the following article, the main problems that engage the attention of the gas industry, and indicates especially the importance of markets for coal tar products, the home production of motor spirit, and the possibilities that appear to lie in the Bergius process.*

It is evident that during the last few decades the average amount of coal used in a normal household has undergone a definite decrease owing mainly to the more widespread use of gas cookers, fires and other appliances. This state of affairs is, of course, very acceptable to the gas industry, but it is also of great importance to the populations of all our large towns because of the influence it has upon the elimination of smoke from our atmosphere. At the same time, it must be realised that in many urban districts there has been a great increase in the concentration of population, with the result that notwithstanding the increased usage of gas, there has been no decrease in the consumption of raw coal for domestic purposes.

### Eliminating Smoke

It is probable that the next big contribution of the gas industry towards the elimination of smoke from our cities will be the more general substitution of solid smokeless coke for coal. Much has been written about low temperature carbonisation, and attempts are still being made to produce low temperature fuels on a profitable scale. Whatever may be the ultimate result of all this work, it is clear at any rate that it has focussed more attention upon the possibilities of using high temperature coke for domestic purposes.

During the last year or two several types of grate have been introduced which may be suitably installed in any domestic apartment, and, particularly having regard to the fact that some of these grates are equipped with means for igniting the coke fire by means of gas, it is obviously a matter of great interest to the gas industry to see that these fires are supplied with a suitable quality of coke.

It may also be noted that the elimination of the coal consuming kitchen range, and its substitution by the boiler, fired with graded coke, is also proceeding steadily, and it is anticipated that this type of appliance will become still more popular when more experience is gained relative to the exact size of coke which should be used with each pattern of apparatus.

These advances in the use of coke for domestic purposes all indicate the desirability of a very close study of the choice of coal used for carbonisation. The gas industry is watching the coal survey work of the Department of Scientific and Industrial Research in the different seams and mining areas of the United Kingdom with much interest. The information collected from these surveys must be valuable in enabling the various consumers of coal to decide which coals are most suited to their purpose. This is a matter of national interest since it should result generally in increased industrial efficiency and economy. For the gas industry it may mean that the field of suitable coals will be widened.

### The Blending Problem

Considerations regarding the purchase of coal on the one hand, and the desired properties of the coke product on the other, lead one continually to study the possibilities of blending different coals before carbonisation. This process, possibly combined with the process of briquetting the blend, still presents great attractions to the carbonising industries.

Hitherto it has made slow progress in the gas industry owing to the difficulty of adapting existing coal handling and coal storage plants. It is, however, certain that still more attention should be given to the design of these plants in order that advantage may be taken of the improvements which may be effected not only in the quality of the resultant coke but also in the economics of the carbonising process itself.

The question of the supply of gas over large areas by means of a network of mains linking up a number of gasworks and coke ovens has been the subject of much discussion in the last year or two, and, as is well known, it led to the appointment of a departmental committee. The report of this committee, published this year, expresses the opinion that such a network is both practicable and desirable in such highly industrial areas as South Yorkshire. The report also expresses the view that if an expanding market for gas is to be found, the charges for gas for industrial operations should be based not only on the commodity value of the gas but also on the service given, and, if possible, the load factor. It is probable that during the coming year attempts will be made to give legislative effect to some of the Committee's recommendations.

The declining revenue from by-products is still a matter of concern to the industry. I referred to the subject of ammonia recovery in these pages last year, and it still appears that the most useful work which can be taken in hand immediately in most gasworks consists in improvements in gas condensation and gas washing conditions. No new process of striking novelty has appeared which leads the industry to look to it for any immediate commercial solution of its ammonia problem.

### The Market for Tar Products

The principal market at the present time in this country for tar products is for road making purposes, and in this connection it may be noted that revised specifications for this product have been issued. The increase in the amount and weight of traffic on the roads demands a more viscous tar than was formerly used. Progress in the production of suitable tars has been made in several directions, particularly with regard to the blending of different tars which alone would not comply with the required specification.

There is evidence of a considerable revival of interest in the use of coal tar oils in internal combustion engines of the Diesel type. Before the war such oils were used in Germany to the exclusion of petroleum oils, and during the war they were used to a considerable extent in this country, but the petroleum oils have now made considerable inroads into this market. The manufacturers and users of large Diesel engines realise, however, that it is in their own interests to secure a supply of fuel produced from our national raw material—coal.

The problem of the home production of motor spirit is one of primary importance, as may be demonstrated by reference to the fact that each year we import about 810 million gallons, whilst only some 30 or 40 million gallons of benzol are produced in this country. While the carbonising industries might pay more attention to the efficiency of benzol recovery with appreciable advantage to themselves, it should be realised that with

the existing carbonising processes we can hope to produce only a small proportion of the total amount of motor spirit consumed in this country. If the whole of the gas produced in the United Kingdom was treated for the recovery of benzol the increased production would amount to about 80 million gallons per annum. This would still leave about 730 million gallons of motor spirit which it would be necessary to import.

#### Conversion of Heavy Oils

The technical literature of the past year shows that remarkable advances are being made in the conversion of heavy oils, including coal tar oils of high boiling point, into motor spirit by the action of hydrogen at high pressure in the presence of suitable catalysts. We may suppose that by such a process it would be possible to produce an extra three gallons of motor spirit per ton of coal from the tar produced during the carbonisation of coal in the established high temperature processes of to-day. If, however, the whole of the present production of tar were treated in this way, the additional amount of spirit produced would only be of the order of 110 million gallons per annum. Again, this is a quantity of no small importance to the carbonising and tar distillation industries, but if, as a nation, we produce this, we are still faced with the necessity of importing about 620 million gallons of motor spirit.

The total amount of coal carbonised is of course governed by the demand for the primary products, gas or coke. Any proposal to increase the supply of home produced oil by the carbonisation of extra coal, even if coupled with the hydrogenation of the tar produced, would create difficulties, at least temporarily, in connection with the disposal of the coke and gas produced.

Production of motor spirit in such quantities as are required can only be secured by some other process of coal treatment.

#### The Bergius Process

The Bergius process for the production of oils from coal by the action upon it of hydrogen under pressure yields much larger amounts of oils than any other method of coal treatment. By this process it is possible, if the oil produced is distilled and the residue again hydrogenated, to produce yields of motor spirit from English coals of the order of fifty gallons per ton.

In addition to the reduction of imports of motor spirit, any such process would be decidedly beneficial to the coal-mining industry by reason of the extra coal which would be required. It is calculated that the extra coal which would be necessary to produce the balance of 620 million gallons of motor spirit by carbonisation, and the coal required for the production of the necessary hydrogen, would amount to some 20 million tons per annum, which represents over 50 per cent. of the total coal carbonised in the United Kingdom for gas manufacture and metallurgical coke. Such an increased output must have a decided influence on the economics of the coal-mining industry and on the unemployment problem. The financial soundness of the coal hydrogenation process cannot be ascertained with any reasonable degree of accuracy at present, as no figures are available for the costing of such a process working continuously on the large scale, but it is certainly a development which should not be neglected and possibly one which might be expedited if means could be found for stabilising the selling price of motor spirit derived from coal.

## Conditions in the Heavy Chemical Industry in 1930

By P. Parrish, A.I.C., M.I.Chem.E., M.I.Gas.E.

*Mr. Parrish, who is recognised as one of the highest technical authorities in certain branches of chemical production, once more reviews the general conditions in the heavy chemical industry, dealing especially with problems affecting by-product and synthetic ammonia, sulphuric acid, and by-products of coal carbonisation in general.*

No review of the conditions in the heavy chemical industry in 1930 can be made without immediate reference to the problem of trade depression and unemployment throughout the world—a matter uppermost in all minds.

How far this is due to over-production or the instability of gold as a standard of value remains to be seen. But the grave warning of Lord d'Abernon cannot lightly be ignored. When, on the one hand, there is a vast volume of production, and, on the other, millions of men insufficiently supplied with the requirements of life, such as food and clothing, Lord d'Abernon's conclusion that failure proceeds from inadequate facilities of circulation and exchange rather than from excessive ability to produce, cannot well be resisted.

#### Synthetic Ammonia Industry

Fertilisers, particularly nitrogenous fertilisers, have not escaped the world-wide depression of markets and prices. It was remarked last year that the danger with the synthetic ammonia industry, regarding it in its widest sense, was that productive capacity was likely to outrun purchasing power, with a possible resort to keen-cut competition, and that savings in production may well be lost in expensive propaganda.

Apart from productive capacity, as foreshadowed, having exceeded purchasing power, there has been a universal agricultural crisis, which has rendered the situation more difficult and perplexing.

An agreement between European producers of synthetic ammonia and Chilean nitrate interests, reached in Paris, provides for the regulation of prices, production, and allocation of markets between different countries. Each producer is to retain his home market, and export trade will be governed by quotas. France is the only country outside the agreement. The anti-trust laws prevent the United States from being a party to the arrangement. It is not improbable, however, that she will restrict her production. The output restrictive scheme will affect a certain number of works; it may even result temporarily in the cessation of operation at certain factories. The compensation fund of three million pounds, constituted by contributions of three-quarters of a million

from the Chilean producers (Cosana), and the remaining two and a quarter millions from the synthetic ammonia producers, has been established.

The Chilean nitrate industry appears to have emerged with improved prospects. She is expected to observe a price level, but otherwise there is no restriction of production. A wider application of the Guggenheim process, and the general rationalisation of this industry cannot prove other than advantageous.

The synthetic ammonia industry is bound to feel the effects of restriction, and will naturally discourage present and prospective competition. It is felt that this has been pursued in some cases in a rather disingenuous way.

Colonel G. P. Pollitt, of I.C.I., presented an interesting paper to the Second World Power Conference, concerning the synthetic ammonia industry. It is a contribution replete with interesting facts and figures, which may be regarded in many respects as authoritative. Where comparisons with other producers are instituted, it is less convincing. Indeed, certain figures have been challenged, particularly the suggestion that hydrogen from coke oven gas cannot compete successfully with that from water gas. Careful consideration of the facts reveals a balance in favour of coke oven hydrogen.

It is well, in a review of this character, to give the original and revised figures, the latter of which are generally accepted.

(1) COLONEL POLLITT'S FIGURES, BASED ON ONE TON OF NITROGEN.

|  | Original figures.           | Revised figures. |
|--|-----------------------------|------------------|
| Cyanamide process .....                      | 12.72 tons coal equivalent. | 12.72 tons.      |
| Bosch synthesis .....                        | 7.27 do.                    | 7.27 tons.       |
| Synthesis using coke oven gas hydrogen ..... | 6.27 do.                    | 3.48 tons.       |

(2) COLONEL POLLITT'S FIGURES OF CAPITAL COSTS.

| Source of hydrogen. | Plant. | Buildings. | Total. |
|---------------------|--------|------------|--------|
| Electrolytic .....  | 40.8   | 5.2        | 46.0   |
| Water gas .....     | 30.5   | 4.6        | 35.1   |
| Coke oven gas ..... | 29.0   | 4.0        | 33.0   |

N.B.—The coke oven gas figure needs to be amended to £15.2 per metric ton/year of ammonia, all included.

## (3) COLONEL POLLITT'S FIGURES OF WORKING COST OF MANUFACTURE OF ONE METRIC TON OF AMMONIA, IN THE FORM OF 25 PER CENT. LIQUOR.

| Source of hydrogen.                              | Cost of ammonia<br>£ per metric ton. |
|--|--------------------------------------|
| Electrolysis, (a) H.T., A.C., at 0.05d. per kwh. | 7.0                                  |
| (b) do. 0.10d. do.                               | 9.3                                  |
| (c) do. 0.15d. do.                               | 11.7                                 |
| Coke oven gas .....                              | 9.0                                  |
| Water gas .....                                  | 7.2                                  |

N.B.—The coke oven gas figure needs to be amended to £6.0 per metric ton of ammonia/year.

The Mont Cenis process, working at lower pressures and temperatures, has established itself in Holland, Germany, France, America and other countries. It is a process that has caused some concern to the I.G., who earlier in the year purchased the original Mont Cenis factory. A general view of the Mont Cenis plant, and of the sulphate stores, is given in Figs. 1 and 2.

From statements which have been made, it is felt that the capital cost of the Billingham synthetic ammonia plant is high when contrasted with German ammonia synthesis plants using hydrogen from coke oven gas.

Speculation has been rife concerning the cost of manufacturing ammonium sulphate (25.5 per cent.  $\text{NH}_3$ ) per ton, net naked in the stores, on the basis of Colonel Pollitt's figures of capital cost, and works cost of manufacturing ammonia, etc., by the anhydrite process. A figure of £5.5s. per ton is suggested as covering all manufacturing charges, supervision, depreciation at  $7\frac{1}{2}$  per cent., rates, insurance, and establishment charges. With the present selling price of ammonium sulphate this leaves a satisfactory margin. It must not, however, be overlooked that the output restrictive scheme already referred to imposes a heavy penalty in the shape of inactive capital charges, (excluded from the foregoing cost per ton figure), variously estimated at from £2 to £4 per ton of sulphate, according to the location of the works.

The number of the new catalysts and methods of preparation and activation is fast diminishing. Some attention is being given to the more economic release of ammonia from solutions of 25 per cent. ammonia. The Fauser process appears to be gaining in popularity. Plants of a daily capacity of 215 metric tons are now operating, and six additional plants are being built, of 640 metric tons capacity, bringing the total daily capacity to 855 tons of ammonia. The situation to many appears paradoxical. There are output restrictive schemes on the one hand, obviously imperative, and yet continued erection of synthetic ammonia plants on the other.

#### Scientific Utilisation of Coal

Reference to the patent literature of the year reveals that the hydrogenation of coal, the distillation and cracking of coal oil and low temperature tars, still continue to receive attention. Pending some successful development on a commercial basis, the economics of low temperature carbonisation must remain uncertain.

Sir David Milne-Watson has indicated that it is probable that the smokeless fuel of the future will be manufactured from selected, prepared and blended coals by high temperature processes. Sir David further declares that "the proper organisation of the carbonising industries can produce 12 per cent. of the motor spirit and 25 per cent. of other imported oils."

A fundamental study of the products obtained on oxidising coal itself, or the residue remaining after extraction with benzene under pressure, has revealed that these are largely a mixture of benzene and carboxylic acids. The significance of the discovery must be considered in relation to the large proportion of benzenoid compounds in the form of phenols and in particular cresols produced on carbonising coal at low temperatures. The high proportion of phenols reduces the value of the distillates produced, and creates problems for economic utilisation that still await solution.

Professor Bone's study shows that brown coals, lignites, semi-bituminous, coking and non-coking, and some bituminous coals, practically all give the same yields of organic acids on oxidation. The raw material of coal must have been collected apart from oxygen under water. It is suggested that coal is no mere residue, but a synthetic re-wrought material similar to synthetic bakelite, in which phenol and its homologues are condensed with formaldehyde and with amino bodies. This is an amplification of Mr. E. V. Evans's previously expressed description that coal was a dried jelly—a colloidal body in a dried state, which may be re-dispersed when treated with suitable agents under appropriate conditions.

Our conception of coal is rapidly widening, and further fundamental studies cannot but prove helpful to all concerned.

#### Sulphuric Acid

The demand for lower prices for this mineral acid cannot be disregarded. The successive yearly reduction in the price of ammonium sulphate, and the lower prices realised for calcium superphosphate must at least cause manufacturers to think. On the other hand, with the present low percentage production at sulphuric acid works (of the order of 60 per cent.), is the stimulus such as to overcome the natural inertia? Yet sulphuric acid manufacturers must not be oblivious to the trend of events. When gasworks and coke ovens are seriously advised to use ammoniacal liquor for the quenching of coke (in other words, to destroy it rather than utilise it more efficiently and more rationally), this country's annual demand for acid from the source in question is at once menaced to the tune of 450,000 tons.

In other directions proposals are being made which cannot but affect the demand for sulphuric acid.

It was remarked last year that the equipment and operation of mechanical pyrites burners can be improved considerably by the adoption of infinitely variable gears, by securing increased flexibility by using air for cooling, and by providing cooling arms in addition to rabble arms in the hottest beds. Results of the operation of mechanical burners equipped in



FIG. 1. MONT CENIS SYNTHETIC AMMONIA FACTORY.



FIG. 2. SULPHATE STORE WITH COLLECTING AND BAGGING EQUIPMENT.

the foregoing way should soon be available. Attempts to secure simultaneous combustion and sintering of certain sulphur-containing ores are proceeding in modified forms of sintering plant.

Further attention has been given to the vanadium type of catalysts, but there is still an appreciable difference in the capital cost of a contact plant and a chamber plant of modern type, the advantage being in favour of the latter.

The Gaillard-Parrish system of sulphuric acid manufacture continues to extend. Fig. 3 shows the construction of a typical chamber. There is more in the liquid phase system than has hitherto been supposed. Apart from the removal of 19 to 20 per cent. of the heat entering and generated in the chambers, liquid phase working possesses marked advantages, and reduces the space-time factor materially. The surface temperature of the lead of the producing chambers need not exceed 30° C. (top portion) and 45° C. (bottom portion), and the temperatures within such chambers range from 62° to 56° C. What this means in the shape of longer life of the lead can best be appraised by those who are working the old type of chamber plant, at 12 to 14 cubic feet of chamber space per lb. of sulphur, with temperatures in the leading chambers ranging from 105° to 80° C.

A marked economy of nitrate of soda can be effected by feeding the Gay Lussac towers with acid of d 1.76-1.79. The trouble incidental to the use of acid of this strength may be overcome by cooling and drying the gases before entering the tower. It is suggested that the second Gay Lussac should be used for this purpose. When it is known that Glover acid can be produced at the strength indicated by a modified arrangement of inlet to the tower, the desirability of operating in this way seems evident.

Increasing attention is being devoted to the recovery of sulphur from pyrites by heat, and equally to the treatment of sulphur dioxide in smelter and roaster gases, for the recovery of this element. In one such process (B.P. 328,029), the General Chemical Co., of America, propose to pass gases containing sulphur dioxide through a bed of hot carbonaceous material, whereby sulphur, carbon oxysulphide and hydrogen sulphide are formed. The gases are then cooled to 120°, to effect deposition of the sulphur, and the residual gases, together with more sulphur dioxide, are passed over a catalyst, for example bauxite, or activated carbon, heated to 200-300° to effect complete conversion of the sulphur compounds to free sulphur.

#### By-Product Ammonia

With successive yearly reductions in the selling price of ammonium sulphate, naturally, the situation here is acute. The Ammonia Sub-Committee of the Institution of Gas Engineers recently completed its survey of the past twelve months' work, and a reasoned summary of the position was prepared by two of its members, Dr. E. W. Smith and Mr. H. Hollings, M.Sc.

It is suggested that gasworks improvements in condensation and distillation conditions along the lines already indicated represent the most useful work that may be taken in hand at once, that consideration should be given to the organisation

of district co-operative schemes for the treatment of concentrated gas liquor, and equally, to the possibility of making the gas industry self-supporting in respect of sulphuric acid.

The writer has made similar suggestions in his annual reviews during the last four or five years. Some resolute and determined action, on sound and approved lines, is now essential. Due regard must be given to the strategic points at which such centrally-situated works should be erected.

The use of anhydrite in the manufacture of ammonium sulphate, so as to place such centrally-situated works, operating co-operative schemes, on a competitive basis with synthetic works, has been studied, and a contribution is published as part of the third report of the Ammonia Sub-Committee. In the report in question it is pointed out that a concentration of ammonium sulphate up to 40 per cent. is attainable. It should be noted, however, that evaporation costs can be obviated if one works on lines which were already known, but which have since been patented by O. Piette (B.P. 327,488). Essentially, what is provided for is this: Ammonium sulphate is precipitated from a cold saturated solution by ammonia. The mother liquor resulting is pumped to a still, and some of the ammonia is released by the application of steam. The cooled liquor is treated successively with carbon dioxide and ground anhydrite, or precipitated calcium sulphate. After filtration, the ammonium sulphate is returned to the saturator for further precipitation.

This cycle of operations is continued, and precipitation of ammonium sulphate is effected in the saturator without the use of steam for evaporation. The only steam employed is that required for releasing the excess ammonia from the ammoniacal mother liquor. Advantageous as this method of working undoubtedly is, it is doubted whether a satisfactory elongated crystal can be obtained. Other provisions and equipment are called for if this process is to produce ammonium sulphate that can compete successfully with the best products on the market.

Attention has been given to the design and operation of concentrated gas liquor plants, and it is now possible to operate these without labour, by adopting a series of special sensitive temperature and pressure regulators, placed at four cardinal points.

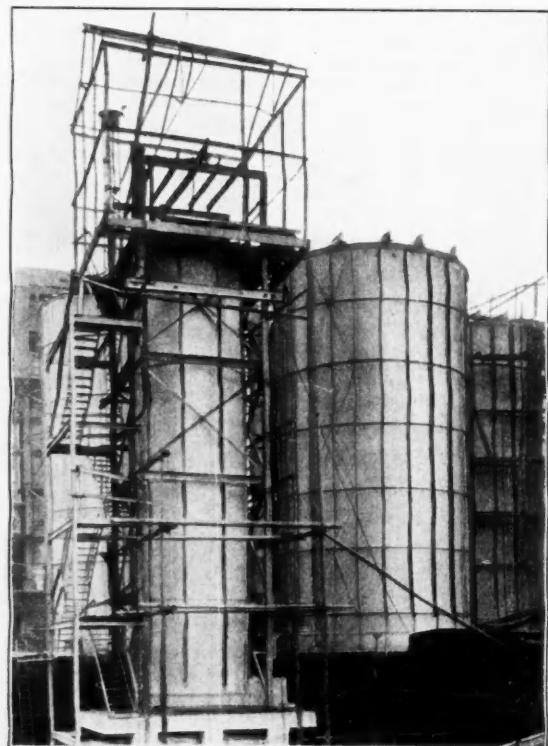


FIG. 3. GAILLARD-PARRISH SULPHURIC ACID PLANT UNDER CONSTRUCTION.

Thus the gas industry is cheapening in a sensible way the cost of production of concentrated gas liquor—a product approaching the synthetic one, but containing sulphides of ammonium, which in some respects are an advantage: in addition, reasonably pure sulphur can easily be recovered by special means.

The production of ammonium bicarbonate direct from gas has been receiving attention during the last twelve to eighteen months, and there may be further developments in this connection. Known methods can be applied for reducing the volatility of this product to a minimum.

#### Alkali

Works in this country removed from brine wells are now electrolysing solutions of common salt, and are producing caustic soda, and liquid chlorine, and utilising the hydrogen released to advantage. Cheap power plays an important rôle in connection with any such development.

The ammonia-soda process still runs hand-in-hand with the electrolytic method, the limitation in the application of the latter on a large scale being the inability to find a suitable market for the disposal of chlorine.

A modification of the Solvay process, which works throughout with solutions saturated with respect to ammonium chloride, has been noted during the year. Ammonium bicarbonate is added to a solution containing 20% of sodium nitrate, and saturated with sodium and ammonium chlorides. The resulting sodium bicarbonate is filtered off, and sodium chloride added to the warm filtrate. This is then cooled to separate ammonium chloride. A works-scale experimental plant is described, in which the ammonium bicarbonate used is prepared from coke oven sources. The sodium bicarbonate produced is calcined and converted into caustic soda. If there were an agricultural demand for ammonium chloride, this method might well be a menace to established alkali works.

The production of hypochlorites in new forms has aroused considerable interest. Concentration is attained in one case to the extent of 38-40% of available chlorine, free from pungent smell, and in the other case containing 65-75% calcium hypochlorite. Many industrial applications can be found for such products.

#### Phosphoric Acid and Associated Products

Considerable advance has been made in the technique of phosphoric acid production, largely directed to the formation of hemi-hydrate crystals, which are permitted to deposit on old crystals. The hemi-hydrate, under ordinary circumstances, would be very fine, but by depositing it on crystals which have already formed, a coarse-grained product, which admits of rapid filtration, is obtained.

Improved methods have also been introduced for the attainment of higher concentrations of phosphoric acid than hitherto. Dependent on the strength of the sulphuric acid, it is possible to obtain phosphoric acid containing 32 per cent.  $P_2O_5$ . Plant of the submerged combustion type has been developed for the evaporation of phosphoric acid this year, and promises to solve many of the difficulties which technicians have hitherto had to face.

Many works producing phosphoric acid are taking advantage of the calcium sulphate precipitate for the manufacture of ammonium sulphate *via* ammonium carbonate, and are also treating the calcium carbonate sludge with nitric acid, in the manufacture of calcium nitrate. The evaporation of this product, no less than its subsequent solidification, after concentration to 80 per cent.  $Ca(NO_3)_2$ , has been the subject of several patents, all directed to overcoming this difficulty.

Improvements have been introduced in the manufacture of nitric acid by the Ostwald process. Many use a mixture of air and oxygen, and recover the oxides of nitrogen in water in suitable towers, using a final wash with sodium carbonate.

Fauser has introduced a method of bubbling the oxides of nitrogen emerging from the conversion chamber through water contained in a series of boilers arranged in cascade form. This method absorbs power, but otherwise has much to commend it. A new works at a coke ovens in France is being erected for the treatment of phosphate rock with nitric acid, in the production of a mixture of calcium nitrate and mono calcium phosphate. At this works it is proposed to use oxygen exclusively with the ammonia in the Ostwald type of converter, for the production of nitric acid, which, being in concentrated form, is passed to a compression pump, and the nitric acid is liquefied in this way. The progress of operations at this works will be watched with interest by all technicians interested in fertilisers.

#### Superphosphate Industry

This industry has been under a cloud during the last twelve months. When there is an agricultural depression there is bound to be a reflex action, and the superphosphate manufacturers invariably suffer. They are still as firmly convinced as ever that concentrated chemical fertilisers are not likely to be as satisfactory as is supposed. They still affirm that their compound manures, constituted of calcium superphosphate, kainit, ammonium sulphate, with additions of bone meal and guano, possess special merits which no chemical fertilisers offer.

They suggest that the soil needs, apart from phosphatic, nitrogenous or potassium elements, bacteria which have usually been provided or the development of which has been aided by the introduction of a little organic matter in their compound fertilisers.

Steps have been taken by a number of important superphosphate manufacturers in Great Britain to improve their plants—acid and superphosphate—in order to reduce costs. They are firmly convinced that calcium superphosphate will be required for many years to come.

What the future has in store it is difficult to predict. Unless some immediate action is taken, on the lines indicated in the earlier part of this contribution, it is feared that emergence from the present depression can only be slow at best.

One cannot at present say that prices have reached bottom, and it is certain that at the moment no organised buying, to replace stocks which one has reason to believe are depleted in many respects, can be discerned.

## Chemical Researches Undertaken by the D.S.I.R.

### Work at the Chemical Research Laboratory

This article contains a brief review of the more important investigations in progress at the Chemical Research Laboratory. Researches of much chemical interest are also being carried out at other stations of the Department. The Associations which have been formed under the scheme of the Department of Scientific and Industrial Research for promoting research in industry also include chemical investigations of considerable importance in their programmes.

#### Synthetic Resins

An indication of the growing importance of synthetic resins in the moulding, electrical and lacquer industries, is furnished by the world's approximate production figures, 13,000 tons of formaldehyde resins having been manufactured in 1926, as against 9,000 tons in 1921. Research has been concerned particularly, but not entirely, in the Chemical Research Laboratory with the important question of dielectric strength.

**Formaldehyde-Phenol Resins.**—The formaldehyde-phenol resins possess valuable heat-hardening properties, combined with high dielectric strength, suffering, however, from the defects of (a) difficulty of producing consistent batches,

(b) the phenomenon of "tracking" under electrical stress, and (c) brittleness when used in lacquers and varnishes. The commercially available "glyptals" are useful in overcoming defects (b) and (c).

Progress towards consistency between batches has been achieved to a marked extent by using a volatile solvent as controlling medium, whereby the temperature of condensation is kept low, and the resin may be obtained at any desired degree of condensation. The progress has been extended to a semi-industrial scale, using 24 lb. of *m*-cresol in a single experiment, the plant involved including a steam-heated condensation vessel, washing and storage tanks, drying vessel, incorporators, hot-air machines, vacuum oven and gas-heated

hydraulic press. The resulting resins have shown breakdown voltage strengths of 700-800 volts/mil. when moulded with wood-meal, and consistent values of approximately 1,600 volts/mil. on single impregnated tissue paper sheets. Phenol itself, less expensive than *m*-cresol, has proved equally satisfactory.

Evidence of the advantage of ammonia over caustic alkalies as condensing agent in producing heat-hardening resins has been supported by electrical tests on laminated boards (cf. T. Hanyu, *Res. Electrotech. Lab.*, Tokio, No. 262, 1929; *Chem. Abs.*, 1930, **24**, 2621). Acidic condensing agents usually lead to permanently fusible and soluble resins (Novolaks).

Various crystalline substances, possibly intermediate stages in resin formation, have been isolated from both alkaline and acidic condensations, hydroxybenzyl alcohols resulting in the former case, while dihydroxydiphenylmethanes predominate under acidic conditions. The isolation from *p*-cresol of a substance of formula  $\text{HO.C}_6\text{H}_3(\text{CH}_3).\text{CH}_2\text{C}_6\text{H}_2(\text{CH}_3).\text{OH}$ .  $\text{CH}_2\text{C}_6\text{H}_3(\text{CH}_3).\text{OH}$  has an important bearing on the hypothetical structure of the Novolak type of resin (cf. Koebner, *Chem. Ztg.*, 1930, **54**, 619).

**Formaldehyde-ketone resins.**—The use of acetone as a solvent in the preparation of the foregoing formaldehyde-phenol resins led to an investigation of its own reactions with the formaldehyde, extended by including its homologues. The formation of resin decreased markedly with rise in the series, diethyl ketone with 8 per cent. caustic soda yielding none, while acetone yielded resin with less than 1 per cent. Various intermediate condensation products have been isolated of the form  $\text{R}(\text{CH}_2\text{OH})_x$ . ( $x$  varying from one to the total number of hydrogen atoms on the two carbon atoms adjacent to the carbonyl group), while dehydration products of these have also been found in the condensations.

The properties of the formaldehyde-acetone resins render them more useful in the sphere of varnishes than in plastics.

#### Low Temperature Tar

During the past five years researches have been in progress on the isolation and identification of chemical constituents of aqueous liquors and tars from low temperature carbonisation of coal, the quantitative analysis of a series of tars produced from various coals and carbonised under different conditions, cracking and hydrogenation of tars and the application of tar products to industrial uses. In the examination of low temperature tars, methods involving distillation, except at low temperature and under greatly diminished pressure, have been replaced by a more modified treatment with solvents, and by this means each of the major constituents, neutral oil, phenols, bases and acids have been separated into crystalloids and resinoids.

**Crystalloids.**—The neutral portion of the tar contains waxes melting over a range up to  $66^\circ$ , unsaturated oils extracted by mercuric acetate and aromatic hydrocarbons, both of the naphthalene and anthracene series. The parent hydrocarbons are present only in relatively small amounts, but mono-, di- and tri-methyl derivatives of anthracene are more abundant. Synthetical methods of preparation of these hydrocarbons affording rigid constitutional proofs have also been developed. The true phenolic fraction is readily removed by extraction with caustic soda containing salt, and the most volatile phenols consist essentially of phenol, the cresols and the xylenols. The volatile bases are mainly tertiary and contain pyridine, mono-, di- and tri-methyl pyridine, quinoline and quinaldine.

**Resinoids.**—The resinoids constitute the least volatile part of each of the major fractions, are insoluble in petroleum and are generally obtained as pale yellow to brown amorphous solids. They possess many of the properties of natural and synthetic resins, and are termed resinenes (neutral), resinols (phenolic), resinamines (basic) and resinic acids (acidic). These substances are promising materials for both scientific and industrial research, and are present in tars obtained from the carbonisation of wood, peat, lignite, cannel and bituminous coal.

**Aqueous Liquors.**—The organic constituents on aqueous liquors consist largely of phenols, amongst which phenol and catechol predominate. Other monohydric phenols are also present along with the two mono-methyl homologues of catechol, and resorcinol and quinol. A fifth type of resin, resinolic acid, constitutes the least volatile portion of the phenols. Small amounts of bases containing aniline and pyridine and a series of fatty acids from formic to *n*-valeric are present.

A series of aqueous liquors has been examined, and it has been shown that the phenolic content varies directly with the oxygen content of the coal.

**Applications.**—Low temperature tar phenols possess considerable germicidal value which reaches a maximum at the fraction boiling at  $140-170^\circ$  under 5 mm. pressure. Chlorination of the phenolic fractions considerably increases the germicidal potency.

"Bakelite" and "Novolak" resins are readily produced by condensation of low temperature tar phenols with formaldehyde in the presence of alkali and acid respectively. Further satisfactory resins can be manufactured direct from that fraction of tar boiling between  $170$  and  $230^\circ$ .

Experiments are also proceeding on the hydrogenation and cracking of low temperature tars for the production of fuels; and on the synthesis of organic intermediates under pressure.

#### High Pressure Syntheses

**High Pressure Chemistry.**—During the past ten years increasing attention has been directed to the use of pressure as a means of facilitating the course of chemical reactions, and research on high pressure syntheses was started at the laboratory in 1926, on the recommendation of the Chemistry Co-ordinating Research Board, whose members were impressed by the possibilities revealed by the work of Patart in France and Badische Anilin und Soda Fabrik in Germany.

The plant required for this investigation was designed and built in the laboratory workshop, and the earliest experiments were carried out with hand compressors. Subsequently motor-driven compressors and circulators were added to the equipment. This plant was first tried out with catalysts of the Patart type (normal or basic zinc chromate) in order to gain skill and confidence in the process. It was thus found that on passing the mixed gases (1 vol.  $\text{CO}$ , 2 vols.  $\text{H}_2$ ) at the rate of 30,000 vols. per hour, measured at N.T.P. over unit volume of such as catalyst at  $380^\circ$  and under 200 atmospheres pressure the hourly production of methyl alcohol was about twice the volume of catalyst space.

The addition of cobalt chromate or nitrate to the foregoing zinc chromate catalyst led to an interesting development, since with the more complex catalyst ethyl alcohol and other higher alcohols made their appearance, although methyl alcohol remained the predominant product. Small amounts of aldehydes and acids were also detected. By the use of mixed cobalt catalysts containing zinc, together with chromium or manganese, the following alcohols have been obtained in addition to methyl and ethyl alcohols: *n*-propyl, *n*-butyl, *iso*-butyl and *n*-amyl alcohols, racemoid *t*-methyl-propylcarbinol  $\text{CH}_3\text{CH}_2\text{CH}(\text{CH}_3)\text{CH}_2\text{OH}$  and *z*-methylpentanol  $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}(\text{CH}_3)\text{CH}_2\text{OH}$ . So far only primary alcohol have been detected. Aldehydic products have been identified as follows:—formaldehyde, acetaldehyde, propaldehyde, *n*-butaldehyde and also certain aldehydals arising from the condensation of the foregoing aldehydes and alcohols. Moreover, the synthetic products contain formic, acetic, propionic and *n*-butyric acids.

The addition of even small proportions of cobalt to copper-manganese oxide catalysts (Audibert type) has a marked effect on the production of ethyl alcohol and its homologues, and a similar result is noticed on replacing the cobalt in these catalysts by iron. The formation of higher alcohols is promoted by alkali hydroxides, the efficiency of which increases with increasing atomic weight to a maximum with rubidium.

#### Corrosion of Metals

Work on the corrosion of metals, whether immersed in salt solutions or exposed to atmospheric attack, is directed largely to the discovery of the factor which controls the rate of corrosion in closely defined conditions. In immersed conditions this factor has been found to be the concentration of an ion in weak potassium chloride and sulphate solutions, and the concentration of oxygen or of hydroxyl in stronger solutions. In atmospheric conditions the controlling factor may be the humidity or the concentration of sulphur dioxide, according to the nature of the atmosphere; in purely marine conditions containing but little sulphur-bearing gas the presence of sodium chloride may be the most important factor. Carbon dioxide, which is often supposed to be the most destructive agent in the atmosphere, exerts relatively little effect on the speed of attack.

Hydrogen evolution due to the interaction of water or salt solution with metals such as zinc or steel has been found to be

of greater importance than is generally supposed: for instance, the proportion of zinc corrosion due to evolution of hydrogen increases with concentration of potassium chloride, and with 2N solutions amounts to 17.4 per cent. of the total corrosion. With mild steel, up to 5 per cent. of the total corrosion may be due to hydrogen evolution, at a temperature as low as 25° C. It is difficult, and sometimes impossible, to detect visually any sign of the evolution of the gas, and the method used for its determination has been combustion on a platinum wire in a closed space, followed by observations on pressure.

An interesting similarity has been found between the chemical composition of the ultimate products of corrosive action and that of certain minerals. For instance, the green patina on old copper roofs contain constituents of similar composition to that of the minerals brochantite and atacamite, of which the co-ordination formulae are, respectively,  $[\text{Cu} \{(\text{OH})_2 \text{Cu}\}_3 \text{SO}_4]$  and  $[\text{Cu} \{(\text{OH})_2 \text{Cu}\}_3 \text{Cl}_2]$ . Complete agreement with these formulae has been realised in corrosion products after 70 years' exposure and upwards; after shorter periods the basicity of the product is in a lower ratio than that of the minerals. Among the corrosion products of zinc exposed to strong potassium chloride solutions beneath an atmosphere of purified air there is evidence for the presence of a constituent similar to the mineral zincite ( $\text{ZnO}$ ).

Technical methods of protecting metals from corrosion have been studied for the light alloys of aluminium and magnesium. The use of the latter is steadily increasing and is kept in check chiefly by the serious tendency to corrosion, particularly in presence of chlorides; to stop this, much work is being concentrated on methods of protection by means of film-formation. A working plant for the anodic oxidation method of protecting duralumin and similar alloys has been exhibited at the Salon de l'Aviation at Paris, where it has aroused much interest.

#### Chemotherapy

In 1927 a joint Exploratory Committee of the Department of Scientific and Industrial Research and of the Medical Research Council decided that there was need for organised research in Chemotherapy, and accordingly the Medical Research Council set up a permanent Committee to advise them on investigations in this field. To this Committee the Department has nominated three chemical members including the Director of Chemical Research. This work of national importance is a joint effort of several groups of chemists working in different laboratories so that a wide and thorough search for greatly needed drugs and therapeutic agents is now in progress. The contribution of the Chemical Research Laboratory to these researches may be classified under the two following headings:—

1. *Analogues of Bayer 205 or Fournier 309.*—There appears to be no definite law connecting the therapeutic activity of medicaments of the Bayer 205 type and chemical structure, but the activity may depend more on the aggregate effect of the whole molecule rather than on the presence in the molecule of any particular group or arrangement.

In a search for more effective agents, compounds have been prepared in which the terminal aminonaphthalenetrisulphonic acids have been replaced by aminocarbazole di- and trisulphonic acids, disulphonic acids of aminofluorene and aminofluorenone and by dihydrothio-*p*-toluidinesulphonic acid, but so far the effect of these substitutions has not been encouraging.

2. *Organic Derivatives of Arsenic and Antimony.*—Since antimony in organic combination appears to possess specific trypanocidal activity and some curative action in Kala-azar, experiments have been made on the preparation of antimony analogues of the more successful arsenicals. The antimony analogue of tryparsamide (phenylglycine-amido-*p*-arsenic acid) has been under examination. In the more stable meta series, phenylglycine-amido-*m*-stibinic acid and certain allied compounds show a slight trypanocidal effect. The antimony analogue of stovarsol (3-acetylaminio-4-hydroxyphenyl arsionic acid) or more probably its internal dehydration product has also exhibited some activity.

The possibility of a beneficial introduction of arsenic into the fluorene nucleus has been considered and experiment has shown that trypanocidal activity is manifested when an arsionic acid radical is present in a fluorene molecule in conjunction with an amino group.

Experiments have also been made on organic arsenicals produced by condensing atoxyl successively with succinic anhydride and with a base such as ammonia, methylamine,

dimethylamine, piperidine or aniline. Certain of these derivatives have also exhibited definite action on trypanosomes.

In addition to the preparation of antimonials directly applicable to therapeutic tests, our knowledge of the organic chemistry of antimony has been extended among aliphatic derivatives by the production of antimony analogues of the cacodyl group and in the aromatic series by the synthesis of cyclic antimonials analogous to the alkyl- and arylcarbazoles.

#### Water Pollution Research

This research originated from a joint request made to the Department of Scientific and Industrial Research by the Ministry of Health and the Ministry of Agriculture and Fisheries.

During the past two years, experiments have been in progress under the auspices of the Water Pollution Research Board on the base-exchange method of water softening. One of the objects of this work has been to determine the most satisfactory way of carrying out the process, such points having been examined as the effect of varying the rate of flow of water through the bed of base exchange material and the quantity, concentration and time of contact of the salt solution used in regenerating this material. There are two types of base-exchange material in actual industrial use, treated minerals and synthetic products. It appears from the result of the Teddington experiments that with treated minerals the exchange of bases is confined to the outer surface of the particles, whereas with the synthetic materials diffusion to the inner surfaces or into the mass of the gel is an important factor. This study of the base-exchange process has also been extended to the case of waters rich in magnesium.

Disintegration of the base-exchange materials and consequent contamination of the softened waters by silica has been investigated. At the rate of flow employed normally in water softening, the silica content of the water is not increased seriously and is certainly not greater than that often encountered in untreated waters.

Some experiments have also been made on the effect of excessive amounts of sodium chloride on the removal of hardness from natural waters by base-exchange materials. Work has been published on the properties of certain base exchange materials and also on the removal of lead, zinc, copper and tin from drinking water by means of base exchange materials.

In addition to this practical work, a report summarising existing knowledge of the base-exchange or zeolite process for water softening has been compiled and published.

#### General Research

During the present session a paper on the chemistry of acenaphthene has been published under the auspices of the Dyestuffs Industry Development Committee. In this memoir the nitration of the hydrocarbon has been studied under anhydrous and hydrous conditions. A new nitroacenaphthene has been identified and used in the preparation of 2-aminoacenaphthene and 2-hydroxyacenaphthene.

Certain aromatic derivatives of tellurium have been utilised in determining the paracor of this element and a new method has been devised of preparing the cyclic telluro-hydrocarbons.

The fourth of a series of memoirs on *cycloseleno-hydrocarbons* has been published, and it appears from these researches that the tendency to form heterocyclic rings containing selenium is conformable to the strain hypothesis postulated to explain the relative stability of homocyclic systems. These cyclic selenium derivatives afford interesting examples of polar and non-polar compounds and in the case of the four and seven membered rings the monomeric substances are accompanied by polymerides which undergo characteristic chemical changes.

The effect of various co-ordinated addenda on the valencies of the currency metals is still under investigation. Stable complex salts of univalent copper and gold have been prepared and more recently  $\alpha\alpha$ -dihyridylargentate salts have been described in which silver exhibits bivalence.

An examination of the derivatives of diphenyl is in progress, inasmuch as this hydrocarbon being now available in large quantities at a low price is likely in the future to become a starting point in the production of technically important intermediates utilisable in the manufacture of dyes or drugs. In this connexion reference may be made to a general method recently devised for the preparation of phenanthridine derivatives. These compounds will be tested in chemotherapy.

## A Year's Developments in the Solvents Industry

### Notable Progress in Great Britain

The following comprehensive review of developments in the solvents industry is particularly valuable for its careful record of the work proceeding in this important field. Attention may be directed to the achievements in this country of British Industrial Solvents.

THE solvent industry has been less affected than most of the other industries by the adverse trade conditions which have prevailed during the whole year, and the demand for many solvents has remained relatively steady, as new uses and applications have been found for several aliphatic compounds which, only a few years ago, were chemical curiosities not often seen or heard of outside of the lecture room. British chemical manufacturers have become aware in time of the necessity and importance of developing the manufacture of these, and the danger that the United States might gain a dominating position in this field has now disappeared. It is a very hopeful sign that some of the British solvent manufacturers are building with a view to the future. One of the most important applications of solvents is in the production of cellulose ester lacquers, and for this high grade solvents of uniform quality are essential. In this respect British manufacturers have nothing to fear from their foreign competitors. The use of brushing and spraying lacquers is spreading everywhere, and many countries, including several of our colonies, offer promising potential markets for British solvents.

#### Solvents

No new solvents of any importance have made their appearance during the year. A number of papers dealing with the general properties of solvents have been published, several of which present features of interest.

From the report of the Chief Inspector of Factories and Workshops on industrial diseases in 1929 it appears evident that with the possible exception of acetic anhydride and the halogenated hydrocarbons, none of the solvents usually met with are sufficiently toxic to be a serious danger to those working on their production or using them. This is confirmed in an article by Brightman in the *Industrial Chemist* (1930, **6**, 209). In a recent pamphlet issued by the Factory Department of the Home Office a number of precautions are suggested to manufacturers and users of cellulose ester solutions.

The properties required in solvents to be used in cellulose lacquer manufacture were discussed by Kraus in a series of articles in *Farbe und Lack*, and the value of the alcohols either as solvents or solvent-activators for nitro-cellulose was dealt with by Lichtenberger in two articles in the American paint journal. A comparison of the plasticisers usually employed had been made by Wolff and Rosen, and their results were published in the *Farben Zeitung* (1930, 35, 20 and 64).

An interesting article by F. Small on the flash points of mixed solvents appeared in *Industrial and Engineering Chemistry* (1930, **22**, 893). Dividing the solvents into groups according to their chemical nature, the present position may be summarised as follows:

#### Ketones

It is very satisfactory to see that practically the whole requirements of the home market for acetone are now supplied by British manufacturers, and that the imports of this chemical have almost fallen to zero. With an increasing production in this country it should be possible also to export to the colonies, and European countries such as Spain where there is a big potential demand and where so far the chemical industry has not yet been scientifically developed.

It appears that acetone is now being made in America on a large scale from isopropyl alcohol, which is derived from propylene isolated from natural gas and petroleum cracking gases. This adds a new method to the three usual commercial sources of acetone. It is quite possible that in a few years time the manufacture of acetone by this means may also be realised in this country.

It is interesting to note in this connection that the Bataafsche Petroleum Maatschappij have recently been granted a patent (B.P. 337,536) for the dehydrogenation of isopropyl alcohol by means of a special catalyst. With regard to the other methods, little development has taken place during the year in the production of acetone by fermentation methods. These methods labour under the disadvantage that they

produce either butyl alcohol or ethyl alcohol in addition to acetone.

The early work of Desborough and his co-workers and the more recent results obtained by Dr. H. B. Hutchinson in altering the equilibrium in the butyl alcohol acetone fermentation and the work of Moscovitz in increasing the yields of acetone produced by *B. macerans* have only partially met this difficulty.

An interesting article on the biological aspects of the production of acetone by *B. macerans* was, however, published by Zacharov in *Zent. für Bakt.* (1930, **80**, 204).

Wood distillation acetone is not produced to any appreciable extent in this country, but it appears to have held its own in the U.S.A. In this connection it is stated in an American journal that the babassu nut which grows extensively in Brazil is being used in that country as a source of acetone.

#### Synthetic Production of Acetone

The last and most recent of the methods for producing acetone industrially, namely, the synthetic method, has made considerable progress, and may eventually supplant other methods in many places. One of the plants erected in this country has a capacity of 3,000 tons per year. The raw materials that are being used for the synthesis are ethyl alcohol and acetylene, and to a smaller extent acetaldehyde and acetic acid. Several patents in this field have appeared during the year, but except in matters of detail these do not contribute any appreciable advance on the methods already known and worked. Both the I.G. Farbenindustrie and the Holzerkohllungs Industrie A.G. have patented processes in which a mixture of acetylene with steam and either ethyl alcohol or acetaldehyde is passed over ketonising catalysts. It is very doubtful whether acetylene can be obtained in Great Britain at a price to render processes of this nature economic. They may become, however, very important in those countries where the cost of electric power is more reasonable.

Several patents have been published in which improved methods for the direct conversion of acetic acid into acetone are claimed, but none of these presents any striking novel feature. The most interesting contribution in this field was contained in a paper read by Dr. Malquori at the Bolzano meeting of the Italian Association of Science. In this are described a series of investigations which were carried out to determine whether the difficulties hitherto met with in the production of acetone directly from acetic acid can be overcome by altering the catalyst used and changing the conditions of the reaction. Of all the catalysts tried, magnesium compounds were the most efficient in producing acetone at relatively low temperatures and the temperatures needed for the optimum yield increased with increasing atomic weight of the cations in the catalyst used. The extent of actual surface contact is of great importance in determining the yields obtained.

In an article in the *Revue Générale des Matières Plastiques* (1930, **6**, 189) it is claimed that "ketols," that is, mixtures of higher ketones, are of considerable value as solvents and plasticisers for cellulose lacquers, and several of these products are described.

Two interesting patents on the production of higher ketones have appeared. That of the Commercial Solvents Corporation, U.S. Patent 1,757,830, described the manufacture of higher ketones by the interaction of acetone and olefines under pressure at high temperatures in presence of a catalyst; the patent of the Société des Brevets Lefranc claims that pure ketones can be obtained from relatively impure alkaline earth salts of aliphatic acids by distilling them under defined conditions in presence of lime and sand.

A certain amount of work has been done on the derivatives and condensation products of acetone, and various patents in this field have been taken out, principally in the U.S.A. and Canada. These do not present any special features of interest. The manufacture of one of the most important derivatives of

acetone, namely diacetone alcohol, which is a valuable solvent, particularly for cellulose acetate, has been started in this country.

#### Higher Alcohols

The abnormal drought in the U.S.A. has affected to some extent the production of butyl alcohol by fermentation in that country, and the Commercial Solvents Corporation, who are without doubt the largest producers of fermentation butyl alcohol in the world, have had to substitute part of the maize used as raw material by rye. This change has been effected without disturbing to an appreciable extent either the yield of solvents or the throughput. The production of butyl alcohol and acetone by fermentation is being developed in Soviet Russia, and a factory capable of producing 2,000 tons per year is said to be in operation. A Dutch chemist, Dr. Gerritson, claims to have discovered a new method of producing butyl alcohol by the fermentation of potatoes.

An interesting study on the metabolism of the butyl producing organisms was published by Wilson and Fred in the *Journal of Bacteriology* (1930, **15**, 17) and a comprehensive history of the fermentation appeared in the fermentation symposium of *Industrial and Engineering Chemistry* (1930, **22**, 1163). In a process patented recently (British Patent 328,723) Deutsche Hydrierwerke A.-G., claim to have discovered a fresh organism for producing butyl alcohol and acetone, which is capable not only of fermenting the usual starch-containing mashes, but also molasses and other sugar solutions. The same claims are made by the I.G. Farbenindustrie for another organism in German Patent application J.33,050, which has become open to public inspection. This organism is said to produce 68 per cent. of butyl alcohol, 16 per cent. of isopropyl alcohol, and 16 per cent. of acetone. In British patent 322,629 the same organism is applied to the production of these solvents from hydrolysed peat. In two British patent applications which are not yet accepted (5246/30 and 5247/30), Wertheim and Pollak claim to have improved the butyl alcohol fermentation process by a gradual acclimatisation of butyl producing organisms to increasing amounts of organic acids, particularly lactic acid.

A new development in the production of higher alcohols by fermentation methods has been protected by T. Verhave (B.P. 315,263, etc.). In these the production of 2'-butylene glycol and diacetyl by the fermentation of carbohydrate mashes with *C. Polymyxa* or *A. Aerogenes* is claimed.

The manufacture of *n*-butyl alcohol from ethyl alcohol *via* acetaldehyde and crotonaldehyde is now well established in this country, and British Industrial Solvents have installed a plant capable of producing 1,500 tons per annum. An interesting paper on the electrolytic reduction of crotonaldehyde to butyl alcohol was published by Shima in the *Memoirs of the College of Science, Kyoto* (1930, **13**, 85), in which it is stated that a 50 per cent. yield of *n*-butyl alcohol can be obtained by reducing crotonaldehyde in 20 per cent. sulphuric or acetic acid solution at low temperatures. Although it seems unlikely that a method of this nature will become of much importance industrially, the application of electrolytic methods to organic chemistry is a field worthy of more study than has been given to it hitherto. A patent of Imperial Chemical Industries granted this year (B.P. 335,683) for the electrolytic purification of butyl alcohol obtained from crotonaldehyde indicates that the chemists of this country are commencing investigations in this field. With the exception of another patent of Imperial Chemical Industries (B.P. 321,523), dealing with an improved method of hydrogenating crotonaldehyde, nothing further of much interest has been published in connection with what one may call the classic synthesis of butyl alcohol.

Two patents of interest have appeared on the direct production of butyl alcohol from ethyl alcohol by catalysis. In one of these (Neumann, B.P. 326,812) ethyl alcohol is passed at a relatively low temperature over a mixed catalyst to give a series of reaction products consisting largely of *n*-butyl alcohol, and in the other (Bataafsche Petroleum Maat, B.P. 336,811) the passage of ethyl alcohol under pressure over a catalyst consisting of copper oxide and magnesia, in which the magnesia predominates, is said to give high yields of *n*-butyl alcohol.

Although the production of alcohols and other solvents from

petroleum hydrocarbons and oil cracking gases is still in the experimental stage in this country, important developments on an industrial scale in these industries have taken place in the United States, principally owing to the more favourable conditions in that country as regards the availability of raw materials. An interesting account of the processes of the Sharples Solvent Corporation, who produce amyl alcohols and their derivatives for the pentane fraction of gasoline, appeared in *Industrial and Engineering Chemistry* (1930, **22**, 439). "Pentasol," which is the mixture of alcohols obtained by saponifying the chlorinated pentanes, is now manufactured in large quantities in the U.S.A. and used as a substitute for amyl alcohol recovered from fusel oil.

#### Unsolved Problems

Many of the problems involved in the conversion of oil cracking gases into alcohols still wait for solution, and it is very doubtful whether an appreciable amount of any other alcohol except isopropyl is actually made in this way.

In an article in *Przemysl Chemiczny*, Holzmann and Pilat recommend the use of a mixture of sulphuric and acetic acids as an absorbing medium for olefines, and the same feature occurs in a patent recently granted to the I.C.I. (B.P. 334,228) for the production of isopropyl alcohol and isopropyl acetate from propylene. Another feature of this patent is the use of distillation under reduced pressure to recover the mixed products of the reaction. The use of metals of the platinum group as catalysts for the reaction between olefines and sulphuric acid is claimed by the Bataafsche Petroleum Maat (B.P. 336,604) and according to a patent granted to the I.G. (B.P. 324,987) bismuth and similar metals are effective catalysts for promoting the absorption of propylene and its homologues by dilute acid under pressure.

The addition of ethyl sulphuric acid to the sulphuric acid employed for absorbing higher olefines is said to prevent the formation of polymerisation products, and this is the subject of a patent granted to S. Hunt and others (U.S. 1,744,207).

The direct conversion of olefines into alcohols by passing them with steam under pressure at temperatures above 300° C over an alumina catalyst is claimed in U.S. Patent 1,738,785 (McKee).

According to an article in *Industrial and Engineering Chemistry* (1930, **22**, 513) isopropyl ether obtained *via* isopropyl alcohol from oil cracking gases is available in large quantities in the U.S.A. and is said to be an excellent solvent for oils, fats and waxes. Several of the problems involved in the analysis of these unsaturated gas mixtures are discussed by Davis and Quiggle in the analytical edition of *Industrial and Engineering Chemistry* (1930, **2**, 39).

#### An Important Contribution

The heterogeneous nature of the reaction products usually obtained have been the principal obstacle to the industrial development of the synthesis of higher alcohols from carbon monoxide and hydrogen, consequently one of the most important contributions of the year in this field is that of G. Natta (*Giornale chimica Industriale ed applicata* (1930, **12**, 169)). Professor Natta has found that with a catalyst consisting of smithsonite ignited under conditions to give a porous mass and impregnated with alkaline salts of the lower fatty acids, higher alcohols can be obtained from water gas with only inappreciable amounts of ketones, hydrocarbons, and other undesirable by-products. Another notable feature of this process is that with certain catalysts an unusually high proportion of ethyl alcohol is formed. An explanation of the mechanism of the reactions involved is put forward which to a large extent seems to have been verified by experimental data. The process has been protected by patents in all important countries. A number of other patents have appeared claiming improvements in the production of higher alcohols and other oxygenated compounds from water gas, but none of these present any outstanding feature of interest.

A higher alcohol of a somewhat different type, tetrahydro furfuryl alcohol, has attracted attention during the year and is said to have valuable solvent properties. Two interesting patents dealing with improved methods for its manufacture have appeared, one in this country (British Patent 337,296, Imperial Chemical Industries) and the other

in the U.S.A. (as Patent 1,756,228, Quaker Oats Co.). Both of these deal with improvements in the hydrogenation of furfural.

#### Methanol

The production of methanol synthesised from water gas has increased very considerably during the year, and Russia is the latest country to start production with a proposed output of about 10,000 tons per year. A considerable amount of literature has been published on the mechanism of the changes involved in the reaction, and several of these present features of interest. In an article in *Giornale chimica Industriale ed Applicata* Natta has discussed at length the relation between the activity of catalysts for methanol synthesis and their chemical and physical structure, and has brought forward evidence that the efficacy of zinc oxide as a catalyst is considerably enhanced by the presence of certain impurities which seem to act as promoters. Equilibrium in the synthesis and decomposition of methanol has been studied by Lacy, Dunning and Storch and the results of their investigations appeared in the *Journal of the American Chemical Society* (1930, **52**, 926). Other articles of a similar nature were published by Wettburg and Dodge in *Industrial and Engineering Chemistry*, and by Plotnikov and Ivanov in the Russian *Journal of Chemical Industry*.

A very large number of patents relating to improvements in methanol synthesis have appeared but very few of them seem to constitute any real advance in the art. A patent granted to Natta (B.P. 330919) claims the use of smithsonite calcined under conditions to give a porous mass as an efficient catalyst for this reaction. Another patent that is perhaps worth mention is that of the Cie de Bethune (French Patent 630680), which claims that methyl alcohol can be obtained by the interaction of methane and oxygen in the presence of a catalyst. At the request of the United States Public Health Service the United States Bureau of Mines has carried out an extensive investigation on the toxic effects of exposure to synthetic methanol.

#### Esters

Several new esters have been manufactured on an industrial scale for the first time in this country during the year. Among these may be mentioned diethyl tartrate, dibutyl lactate, diamyl lactate and the phthalates of butyl and amyl alcohols. These two latter esters have found application as a substitute for mercury in vacuum pumps and diamyl phthalate has also been found as considerable use as a heat transferring medium in place of the oil usually employed for such work. Although not yet made in this country diethyl carbonate is being produced in appreciable quantities in the United States and is said to possess considerable value as a solvent of medium boiling point for nitrocellulose. A product containing 90 per cent. of ester is marketed under the name of "diatol." Phthalic esters are also being manufactured from "pentasol" and the Selden Chemical Company have obtained a patent for improvements in the production of these (U.S. Patent 1764022).

The use and production of  $\alpha$ -hydroxy-isobutyric esters

is claimed in U.S. patent 1775636 Commercial Solvents Corporation. These esters are said to be excellent solvents for nitrocellulose. Several other patents have been published for improvements in ester manufacture but none of them contain any outstanding feature of interest and very little of interest has appeared in the literature on the chemistry of esters. The most interesting paper is perhaps that of Alinari on the eutectic mixture of alcohols and acetic esters which was published in the *Annali di Chimica Applicata* (1930, **20**, 59).

#### Miscellaneous Solvents

An interesting article on the cyclo hexanol series of solvents appeared in THE CHEMICAL AGE (1930, **22**, 601), in which the numerous applications of this series are described. Dichloroethyl ether is now being produced in relatively large amounts in the U.S.A. and is said to combine the solvent properties of ether and ethylene dichloride. Although not in itself a solvent for cellulose esters it becomes an active solvent when mixed with 10 to 30 per cent. of ethyl alcohol. Diethyl acetal is a latent solvent of the same class and the use of mixtures of this with anhydrous ethyl alcohol is claimed in the British patent application of the United States Industrial Alcohol Co., No. 18660, now open to public inspection.

A large amount of literature has been published during the year on the halogenated derivatives of ethylene. Two interesting papers by Bahr and Zieler in the *Zeitschrift fur Angewandte Chemie* describe the optimum conditions for the production of ethylene dichloride and for the subsequent conversion of this into ethylene glycol. The results of investigations on the synthesis of ethylene chlorhydrin from ethylene and hypochlorous acid were published in the *Giornale di Chimica Industriale ed applicata* 1930, **12**, 283, and the optimum conditions of the reaction have been determined. The value of ethylene dichloride as a component of lacquer solvents was discussed by Frezer and Reid in *Industrial and Engineering Chemistry* (1930, **22**, 604) and in another article in the same journal, Russ (1930, **22**, 844) described the application of ethylene oxide and ethylene dichloride as fumigants. Very considerable amounts of ethylene dibromide are being produced in Germany and exported to the U.S.A. for use as an anti-detonant. A very large number of patents have also been granted for the production of these halogenated compounds and their derivatives, but none of them introduce any radical modification of the well known methods of preparation, and all of them rely on matters of detail for novelty.

Solvents of the cellosolve type are being produced and used to a considerable extent in the U.S.A. and to some extent in this country, but no patents nor articles of real interest have been published on these glycol esters and ethers.

#### Solvent Recovery

With the exception of the papers read at the Institution of Chemical Engineers and referred to in the last annual review number, practically nothing of interest has been published during the year in connection with solvent recovery. A few patents on this subject have been granted but all of them only refer to quite minor details of process or plant design.

## The British Synthetic Fertiliser Industry Progress at Home and Overseas

*The progress made at home and abroad in the important field of synthetic fertilisers is clearly set out in the following article by an expert authority on the subject.*

In last year's review, the growth of the synthetic nitrogen industry at the Billingham factory of Imperial Chemical Industries, Ltd. was briefly described. During the past year production has continued and the tonnage of ammonium sulphate despatched from Billingham has increased by approximately 100 per cent. The by-product production of ammonium sulphate more than suffices for the requirements of the home market, so that the bulk of the synthetic production has been shipped abroad. Spain and the Far Eastern countries—China, Japan, and India—provide the largest markets, but there have been significant increases in some of the smaller markets, notably Australasia, Palestine, and the East Indies, in all of which consumption has more than doubled.

At Billingham intensive research has been carried out with the object of improving the physical condition of sulphate of

ammonia and reducing the tendency of this material to cake during storage and during shipment overseas. Though this work is not yet complete, considerable progress has been made along several lines of attack, and there are definite indications that in the very near future methods will be available, the adoption of which will minimise the tendency of sulphate of ammonia to cake.

#### Nitro-Chalk

The production of Nitro-Chalk, which was first marketed in granular form in 1929, has continued, and this year a new plant with double the capacity of the original one has been brought into operation. Despatches have increased by approximately 150 per cent., the most notable increases in consumption being in the home market, Spain, and Egypt. This fertiliser is made in granular form by spraying a suspension of

chalk in a highly concentrated solution of ammonium nitrate down a tall tower. This tower on the new plant is 250 ft. high and is one of the tallest structures on the Tees-side.

#### Ammonium Phosphate

The manufacture of ammonium phosphate, which was started at Billingham towards the close of 1929, was referred to in last year's article. This year the operation of this plant has been modified to enable the production of a wider range of nitrogen-phosphoric acid fertilisers. During the coming season four grades will be introduced, with the following analyses:—

| Type.   | N. P <sub>2</sub> O <sub>5</sub> |               |        |
|---------|----------------------------------|---------------|--------|
|         | Per cent.                        | Water Soluble | Ratio. |
| A. .... | 14.3                             | 43            | 1 : 3  |
| B. .... | 18.0                             | 18.0          | 1 : 1  |
| C. .... | 16.0                             | 32.0          | 1 : 2  |
| D. .... | 12.3                             | 56.5          | 1 : 4½ |

In these fertilisers all the nitrogen and phosphoric acid is water soluble.

#### Complete Fertilisers

The effectiveness of any "single plant food" fertiliser is dependent on there being available to the plant a sufficiency of the other two major plant foods. This in general implies the use of fertilisers containing all three major plant foods in balanced proportion—i.e., complete fertilisers. To meet this need arrangements have been made at Billingham to manufacture concentrated complete fertilisers in a granular condition, and the necessary plant has been erected. These contain ammonium phosphate, ammonium sulphate and potassium chloride or potassium sulphate. The granulating process ensures the intimate incorporation of all constituents and in each individual granule the N : P : K ratio is the same as in the whole bulk. The granular form ensures perfect physical condition and easy and uniform distribution on the field. Tests have been carried out on standard fertiliser distributors, and it has been demonstrated that these granular fertilisers can be distributed more evenly and at lower rates per acre than the present crystalline and amorphous fertilisers.

During the coming season six granulated concentrated complete fertilisers will be marketed. These fertilisers have been designed to meet the requirements of all crops grown in the United Kingdom: field trials carried out during the past year have proved them to be equally effective as the present grades of mixed fertilisers on the market. They contain approximately twice as much plant food as the mixed fertilisers now commonly in use, and the consequent savings in transport and handling on the farm are apparent. Furthermore, economies in manufacture make it possible to sell them at slightly lower unit prices. On analysis these concentrated complete fertilisers show:—

| N.         | P <sub>2</sub> O <sub>5</sub> |            |           | Potash. |
|------------|-------------------------------|------------|-----------|---------|
|            | Water Soluble.                | Insoluble. | Per cent. |         |
| No. 1 .... | 12.5                          | 12.5       | —         | 15.0    |
| No. 2 .... | 10.4                          | 10.4       | —         | 20.8    |
| No. 4 .... | 10.4                          | 20.8       | —         | 10.4    |
| No. 5 .... | 8.0                           | 16.0       | 5.5       | 16.0    |
| No. 6 .... | 7.5                           | 26.0       | 6.0       | 7.5     |
| No. 7 .... | 6.5                           | 22.5       | 3.0       | 13.0    |

#### Agricultural Research

Jealott's Hill, the Agricultural Research Station of Imperial Chemical Industries, Ltd., has, during the past year, tackled a number of fertiliser problems both in the laboratory and on their 500-acre farm. Many parties of farmers and agricultural authorities visited the station during 1930 and all seemed impressed with the practical importance of the work undertaken. Jealott's Hill is now the central link in the chain of research and advisory work conducted by the company in this country and overseas. During the year several of the senior members of the staff have travelled abroad to increase the station's equipment of first-hand knowledge of the problems which face farmers overseas.

On the Jealott's Hill farm a great number of field experiments have been undertaken. Their purpose, demonstrated in many ways, is to show how fertilisers can best be employed to increase output and reduce costs of production. One trial clearly shows how properly managed grassland will maintain a full head of dairy cows through the year with the minimum of cake feeding.

It was an eye-opener to many who visited the station in the past summer to find that heavy milking cows were doing just as well on intensively managed grassland alone as similar cows did with extra feeding. By this experiment the farmer has been shown how he can save £2 a head in feeding costs through the summer by the intelligent use of fertilisers. The cumulative effect of these and the many other experiments and demonstrations throughout the country has undoubtedly stimulated an entirely new demand for all types of fertiliser for grassland—a demand from which all manufacturers have benefited.

Apart from the herd of dairy shorthorns, there are at Jealott's Hill fattening cattle, a flock of Kerry Hill ewes and Wessex Saddleback pigs, all of which are used for experiments of one kind or another. Many other fertiliser trials are being carried out on cereal crops, the results being recorded with the greatest accuracy. Similar trials are conducted at many farms throughout the country to demonstrate the value of different fertiliser treatments. Particularly interesting this year are the trials with the new concentrated complete fertilisers on sugar beet and potatoes, results of which will shortly be available to the public.

#### The Fertiliser Industry at Home and Abroad

*The following survey of the industry is supplied by The Fertiliser Manufacturers' Association, Ltd., and the International Superphosphate Manufacturers' Association.*

In Great Britain and Ireland it was anticipated that the fertiliser year 1929-1930, which commences in July and ends in June, would not be very satisfactory from the point of view of consumption owing to the very serious position of the agricultural industry. But unfortunately, bad as was the outlook for the farmer at this time a year ago, conditions steadily deteriorated throughout the season, the only relatively bright spot being the cultivation of sugar beet, the acreage of which increased considerably as compared with the preceding season.

Seeing that, on the whole, there was only a relatively small decrease in the total consumption of superphosphate and sulphate of ammonia, it may be assumed that the lesson preached for years to farmers—that in bad times it is a great mistake to reduce orders for fertilisers—has at last had a marked effect. It may not be out of place to emphasise that by reducing fertiliser dressings farmers inevitably reduce their crop in quantity and quality, besides which the fertility of the soil depreciates, and none of these results can possibly be of benefit to the farmer under any circumstances.

The statistics of consumption of sulphate of ammonia during the year 1929-1930 show that the total quantity used by farmers in Great Britain and Ireland was 183,516 tons as compared with 186,710 tons in the preceding year, a reduction of 3,194 tons or 1.7 per cent. England, Wales, and the Channel Islands used 108,441 tons, practically the same quantity as in the preceding year; Ireland 34,483 tons, an increase of nearly 3,600 tons, and Scotland 40,592 tons, a decrease of 6,480 tons. It seems probable that in Ireland particularly the propaganda for intensive cultivation of grassland by repeated dressings of sulphate of ammonia has had good results.

In regard to superphosphate the total quantity manufactured in British and Irish factories during the year 1929-1930 showed an increase of over 19,000 tons; but this merely equalised a decrease of about the same quantity in imports from abroad. The total consumption was 686,300 tons, compared with 724,200 tons in 1928-1929, a reduction of 37,000 tons, or say 5.2 per cent. The whole of the increased quantity of superphosphate manufactured in the home factories went therefore to increase stocks.

The sad straits to which agriculture is now reduced do not augur well for the present year, and if the prospects for the main consuming season, February/May, 1931, are to be judged by the demand during the autumn of 1930, a further set-back in total consumption may be experienced. It is very desirable that all who are in a position to influence the farmer should press home the maxim which experience has taught, "it pays to fertilise."

#### The Position Abroad

The world production of superphosphate for the calendar year 1929 reached the high water mark of 154 million tons,

which was very slightly in excess of the year 1928 figure, but the best estimates of consumption place the total in the neighbourhood of 15,025,000 tons, hence for the world as a whole an increase in stock occurred of over 200,000 tons. Europe, however, exclusive of Russia, made a very much better showing with a 1929 production of about 8,825,000 tons and a consumption of 8,682,000 tons. Europe is a very large exporter of superphosphate to destinations outside its borders, about 230,000 tons being so consigned in 1929; adding this export to the consumption, there is a total sale of 8,912,000 tons, say 87,000 tons in excess of production or an equivalent decrease in stock.

It is much too early to attempt anything other than an approximate estimate of the probable results of 1930, but judging by the reports received from European countries, a decrease which may amount on the whole to between 5 per cent. and 10 per cent. in the consumption is adjudged to be likely, accompanied by an increase in production. The heavy fall which has occurred in the prices of the staple products of agriculture and which has brought in its train an acute economic crisis has undoubtedly caused a contraction in demand, and is likely to bring about a further falling off in 1931. The outlook is, therefore, a very serious one for the superphosphate trade.

The nitrogen industry, which makes up its statistics for fertiliser years, has taken steps to form a convention in an endeavour to regulate production to consumption. In the past two fertiliser years there has been a vast increase in the capacity of the industry to produce which has far outrun the expansion in consumption. Thus in the year 1928-1929 (July to June) 2,113,000 tons of nitrogen were produced in the world, whereas the total consumption was only 1,872,080 tons; the resulting pressure of stocks was accentuated during 1929-1930 when only 1,958,720 tons were consumed against a production of 2,178,400 tons. Of the 1929-1930 nitrogen production 835,000 tons, or say 40 per cent., was in the form of synthetic or by-product sulphate of ammonia, a decrease of about 26,000 tons as compared with the previous year.

The world's agriculture is estimated to have consumed 1,684,000 tons of nitrogen in 1928-1929 and 1,721,000 tons in 1929-1930. Equally with superphosphate it is to be feared that there will be a decrease in consumption of nitrogen during the present fertiliser year, and, having regard to the fact that there are some very large synthetic nitrogen plants just now reaching the production stage, the difficulties which the convention has to face during its first year of existence are of a most formidable character.

## A Review of the Year's Chemical Inventions

By Our Patents Correspondent

*Our Patents Correspondent, in the following article, gives an indication of the general trends of chemical invention during the past year.*

THE British Patent Office receives annually nearly 40,000 applications for patents and approximately some 2,500 of these are intimately concerned with processes employed in industrial chemistry, including metallurgy. The number is, of course, very much larger in the wider field, which includes the processes, based, at least in part on chemical principles, employed in the manufacture of artificial silk, cements, fuels, foods, beverages, glass, leather, etc., and in the bleaching, dyeing, electro-plating, ceramic, photographic, and other arts, and the apparatus and processes employed in distilling, evaporating, drying, filtering, heating, cooling, mixing, and other chemical engineering operations.

It is of interest to note that in the narrower field defined above, with which the present review is in the main concerned, about half of the applications are received from the two giant organisations of I.G. Farbenindustrie Akt.-Ges. and Imperial Chemical Industries, Ltd. Moreover, the number of other industrial interests concerned with this field is quite small, whereas in the wider field the other interests are much more numerous, and are responsible for a considerable majority of the applications.

An outstanding event of the present year was the hearing in the High Court of Justice, occupying some 30 days, of a petition made by Imperial Chemical Industries, Ltd., for revocation of three patents of the I.G. Farbenindustrie Akt.-Ges., as fully reported at the time in THE CHEMICAL AGE. The fact that the patents in question were held to be invalid because the patentees were unable to establish any substantial advantages from the "selections" which constituted their inventions, is of far-reaching importance in relation to chemical patents, as is also the fact that the general principles governing the whole difficult question of what constitutes invention by selection, were fully enunciated by Mr. Justice Maughan.

A few indications are here given of the general trends of invention in some of the more important branches of industrial chemistry. References to individual inventions have been restricted to a few which serve to illustrate such general trends. Numbers of the Specifications referred to are given in parenthesis, together with the volume and page of THE CHEMICAL AGE, where an abstract may be found.

### Destructive Hydrogenation

Research in connection with the destructive hydrogenation of coal, oil, and other carbonaceous materials continues, as for several years past, to contribute a very large number of inventions in this field. Most of these are directed to modifications of the known processes and apparatus by way of improving the circulation of the materials, the control of the pressures

used, the efficiency of the heating systems, and the separation of the products. Choking of the catalyst, especially that due to carbon compounds of high molecular weight, continues to present difficulties, and among the proposals for avoiding it have been the removal of such compounds by condensation or adsorption or their conversion into compounds of lower molecular weight (325,862, Vol. XXII, 422; 332,336, Vol. XXIII, 259), and the addition of an organic liquid of high boiling-point, such as anthracene oil (328,586, Vol. XXIII, 13).

Another undesirable feature, viz., the accumulation of the catalyst in the lower parts of the reaction vessel, has been met by supplying the catalyst in the form of a colloidal dispersion (326,184, Vol. XXII, 442), or using a catalyst of such a density that it remains in permanent suspension in the liquid (328,992, Vol. XXIII, 33).

### Hydrocarbons

In the treatment of hydrocarbons some of the main lines of inventive activity, apart from the cracking processes, have been in the directions of catalytic and other polymerisations and condensations to obtain heavy oils, resins, etc., and catalytic oxidations and dehydrogenations. New polymerisation processes for isoprene, butadiene, and the like, to obtain products for use in the manufacture of rubber substitutes continue to be much in evidence, and other recent polymerisations include that of ethylene to butylene by treatment with boron fluoride in presence of finely-divided nickel and an accelerator such as water or alcohol (326,322, Vol. XXII, 443), of ethylene to benzene by heat and pressure in the absence of a catalyst (331,186, Vol. XXIII, 192), of  $\nu$ -butylene and its homologues to bimolecular and higher polymers (322,102, Vol. XXII, 82), and the polymerisation and hydrogenation of acetylene in successive catalytic operations to obtain hydroaromatic hydrocarbons (334,203, Vol. XXIII, 412). Some condensations of polynuclear hydrocarbons with olefines to obtain resins and viscous oils have also been described (323,100, Vol. XXII, 182; 326,500, Vol. XXII, 467).

Many of the oxidation and dehydrogenation treatments have been for the production from paraffin waxes of higher fatty acids and esters suitable for use as ingredients of boot and floor polishes. Others are directed to the production of carbon black for use in printing inks and in the vulcanisation of rubber. The production of carbon black by other methods is of interest in this connection. Such are, for example, the catalytic decomposition of carbon monoxide under pressure (321,402, Vol. XXII, 31; 327,548, Vol. XXII, 562; 334,190, Vol. XXIII, 412), and the catalytic decomposition of saturated

or unsaturated hydrocarbons or of carbon monoxide in a non-sparking electric field (326,913, Vol. XXII, 494).

Several processes for effecting the catalytic combination of acetylene with water vapour to obtain acetaldehyde may here be noted, the improvement being found in most cases in the use of special catalysts. Such catalysts are, for example, non-volatile mercury compounds, various compounds of tungsten, alumina activated by oxides or sulphides of cadmium or zinc, and zinc oxide activated by a small percentage of a molybdate.

#### Dyes Derived from Anthraquinone

The number, variety, and complexity of the inventions in the dyestuff industry, indicate the exceptionally high standards of research maintained in this field; at the same time they make it impossible to present more than the outlines of a few main developments in a brief review. During the past four or five years the ingenuity of the dyestuff chemist has been increasingly exercised in connection with the anthraquinone derivatives, especially the vat dyes of this type. While the present year has seen a continuation of this preoccupation with the vat dyes of complex molecular structure, much attention has also been given to the comparatively simple substituted anthraquinones which are in many cases suitable for application to acetate silk, and the sulphonated anthraquinone derivatives which are acid wool dyes have not been neglected. Current inventions relating to the mordant dyeing hydroxyanthraquinones of the alizarin type are not much in evidence. No attempt to summarise recent developments in the vast field covered by the vat dyes of the benzanthrone, indanthrone, flavanthrone, pyranthrone, pyrazolanthrone, anthraquinone-acridone and similar series can here be made. A few lines of invention applicable to such dyes in general may, however, be mentioned. These include the separation of ester derivatives from melts containing them, the preparation of dry powders from aqueous pastes of the dyes, the synthesis of anthraquinone derivatives from benzoquinone and 1:4-naphthoquinone derivatives by condensation with 1:3-butadiene (327,128, Vol. XXII, 519), the production of anthraquinone vat dyes containing iodine (327,702 and 327,711, Vol. XXII, 562-3), the production of acid wool dyes from halogenated vat dyes by treatment with sulphites to effect the replacement of halogen by the sulphonate group (332,249, Vol. XXIII, 258), and the purification of anthraquinone vat dyes in general by treatment with hypochlorite and boric acid or another acid of equivalent strength in dilute solution (333,191, Vol. XXIII, 332).

#### Dyeing Acetate Silk

Among the simpler substitution products of anthraquinone, referred to above, as dyes for acetate silk are various *N*-substituted 1:4-diaminoanthraquinones (322,576, Vol. XXII, 125), and 2-alkoxy-1-amino-4-alkyl (or aryl) aminoanthraquinones (323,026, Vol. XXII, 181). Some of the more complex anthraquinone derivatives have also been suggested, *e.g.*, monoaminoanthranthrones (323,750, Vol. XXII, 249), monosulphuric esters of anthrahydroquinones (332,907, Vol. XXIII, 312), and 5:8-di(monomethylamino)-1:2-benzanthraquinone (334,565, Vol. XXIII, 435), and several specifications describe the use of analogous substituted amino derivatives of benzoquinone and naphthoquinones. Another range of vat dyes described as suitable for acetate silk is obtained by treating the anhydride, imide or mono- or di-amide of phthalic acid with ammonia or a primary monamine in presence of iron, nickel, or copper, or their compounds (322,169, Vol. XXII, 101). Compounds containing hydroaromatic nuclei such as the cyclohexyl derivative of 4-amino-1:8-naphthalimide and nitro and azo dyes containing the *α*-tetrahydronaphthalene nucleus have also been suggested, and other interesting types of azo dyes for the same purpose have been mono-, di-, and poly-azo dyes obtainable from diazotized aminonitro compounds containing two aryl nuclei linked in any desired manner (323,792, Vol. XXII, 250), azo dyes having as coupling components various quinolones (327,394, Vol. XXII, 541), azo dyes obtainable by coupling diazotised oxamic acid derivatives of 4:4'-diaminodiphenylmono- or tri-sulphide with suitable components (329,049, Vol. XXIII, 55), and esters of reduced vat dyes containing azo linkages (333,506, Vol. XXIII, 358).

#### Dyeing Viscose

While the dyes having affinity for acetate silk are of numerous types, some of which are indicated above, dyes for viscose

and like regenerated cellulose materials are almost entirely confined to the azo class, and whereas the acetate silk dyes are mainly insoluble unsulphonated compounds applied in dispersed form, the viscose dyes usually contain at least two sulphonate groups and may therefore be applied in aqueous solution. Most of the substantive azo dyes for cotton have a marked affinity for viscose but the number of those which give dyeings in which the colour is evenly spread, without streaky or patchy effects, is strictly limited to a few ranges, and inventors have been trying for some years past to define these ranges in their patent specifications. Each such range may, of course, include hitherto known dyes only or hitherto unknown dyes only or may include both. In the specifications accepted during the present year these ranges have been mainly of disazo and trisazo dyestuffs derived from tetrazotized bases of the 1:3- and 1:4-diaminobenzene, 1:5-naphthylene-diamine, benzidine, thioaniline, diaminodiphenylamine, and diaminodiphenylurea series and sulphonate acids thereof, and having as coupling components in most cases at least one molecular proportion of a 1:8-, 2:8-, or 2:5-aminonaphthol-sulphonate acid or an *N*-substitution product thereof.

#### Other Azo Dyes

There has been continued activity during the year in the production of metal compounds of azo dyes, *i.e.*, azo dyes containing in complex combination one or more mordanting metals, particularly chromium, copper, iron, nickel, and cobalt. These compounds were first introduced some fifteen years ago for dyeing wool, but most of the recent inventions are directed to defining ranges of these colours suitable for dyeing cotton, viscose and other cellulosic fibres or for colouring varnishes or lacquers. Their special value in the lacquer industry appears to be mainly due to their exceptional fastness to light.

In the production of azo dyes on the fibre (ice colours) some new ranges of dyes of the Naphthol AS type, *i.e.*, dyes having as coupling components arylamides of 2:3-oxynaphthoic acid or of acetoacetic acid, have been suggested. In many of these inventions of recent years a selection has been made of particular groups of diazo components in order to ensure various desirable qualities of colour range or fastness in the dyeings. During the present year, however, interest appears to be centred more in the coupling component side of the molecule, and among the arylide components suggested we have, for example, arylamides of 6-amino (or substituted amino)-2:3-oxynaphthoic acid (330,349, Vol. XXIII, 125), 2-(2<sup>1</sup>-oxy-3<sup>1</sup>-naphthyl) amino-5-halogen-1:4-dialkyloxybenzenes (331,247, Vol. XXIII, 192; 332,932, Vol. XXIII, 313), and 4-(2<sup>1</sup>-oxy-3<sup>1</sup>-naphthyl) aminodiphenylamine (332,319, Vol. XXIII, 259).

Stable diazonium compounds for use in the production of azo dyes on the fibre have been described in the form of diazonium fluorsulphonates (321,737, Vol. XXII, 57) and products obtainable by reaction of diazonium compounds with the hexafluoric acids of titanium, aluminium, tin, or antimony, or with the tetrafluoric acids of antimony or zinc (332,227, Vol. XXIII, 258).

#### Wetting, Dispersing, etc. Agents

Apart from the dyes and the dyeing processes one of the main contributions of chemical science to the textile industries is a constant stream of inventions of new wetting-out, cleansing, and emulsifying agents. The prototype of all these "industrial soaps," as they may be collectively designated, is ordinary turkey red oil (sulphonated castor oil). They are for the most part new sulphonate acids of a complex character, and are usually applicable also as dispersing agents for insoluble dyes, especially dyes to be applied to cellulose ester or ether fibres or fabrics, and as pasting agents in textile printing. As typical recent examples may be mentioned products obtainable by sulphonation of a mixture of an aromatic hydrocarbon or a hydroxy derivative thereof and a natural resin, followed by condensation with an aralkyl halide (321,190, Vol. XXII, 12) and products obtainable by condensing furfural or its derivatives with carbocyclic or heterocyclic compounds and sulphonation of the condensation products (322,737, Vol. XXII, 153). A process for sulphonating by the use of gaseous sulphur trioxide has also been described (330,904, Vol. XXIII, 169). Unsulphonated dispersing agents for dyes, pigments, etc., have been suggested in the form of polymerisation products of butadiene or its homologues containing alkyl radicles in the 1- or 1:4-positions (332,557, Vol. XXIII, 291).

### Potassium Nitrate Fertilisers

Several new processes for the conversion of potassium chloride into potassium nitrate are to be noted among recent inventions. In most of these a double decomposition of potassium chloride with nitric acid or with aluminium nitrate or ferric nitrate is effected and the yield and efficiency of the process are increased by provision for recovery and utilisation of the nitrogen oxides and chlorine present in the gases evolved. In a process involving interaction of potassium chloride with calcium nitrate an increased yield is obtained by adding ammonia to the solution before separation of the potassium nitrate (328,272, Vol. XXII, 612). Other inventions of interest in this field relate to the production of mixed fertilisers containing isomorphous mixed crystals of potassium ammonium sulphate nitrate (328,620, Vol. XXIII, 14), the production of potassium carbonate-ammonium nitrate mixtures (329,326, Vol. XXIII, 56), various processes for reacting upon potassium chloride with ammonium nitrate and/or with liquid ammonia, and one in which carbon dioxide and ammonia are passed into an aqueous solution of sodium nitrate and potassium chloride with separation of sodium bicarbonate from the compound fertiliser on account of differences of solubility (331,451, Vol. XXIII, 214).

### Insecticides and Fungicides

An unusually large number of inventions during the present year relates to new substances or preparations stated to have insecticidal or fungicidal value. Substances described as having moth-proofing qualities are particularly notable. Their chemical constitutions are very diverse. Thus we have condensation products of triarylphosphine oxides with phenols (326,137, Vol. XXII, 442), arylsulphonic acid amides substituted in the amino group by alkyl, aryl, or aralkyl groups (324,962, Vol. XXII, 371), various acyldil derivatives of thiourea (326,567, Vol. XXII, 468), phenol-aldehyde condensation products (330,893-4, Vol. XXIII, 169) and many others. Substances described in general terms as insecticides are also of the most varied types, including, *inter alia*, diazoamino compounds, cupric cyanogen compounds, aromatic methoxy-sulphocyanides, aromatic aminosulphonic acid chlorides, and Bz-nitromonohalogenquinazolines. The fungicidal substances are mainly for incorporation in preparations employed in the treatment of seed grain.

### Synthetic Rubber

While some revival of interest in the synthetic production of rubber-like products was indicated by a few specifications for such inventions accepted during the year 1929, the present year has seen a greatly increased activity in this field. In view of the present phenomenally low price of the natural product this increased activity was hardly to be expected. It may perhaps be due in some measure to the time lag between the stimulus afforded by the favourable market conditions for rubber of a few years ago and the inventors' response thereto, and the fact that Patent Specifications accepted during the present year correspond to applications filed on an average some 15 months earlier may partly account for it. Another consideration is doubtless the fact that many of the products and by-products have other valuable uses, *e.g.*, as ingredients of lacquers and in the manufacture of films and thermoplastic materials, and for that reason research in this field has a current economic value apart from its original objective, the production of a synthetic caoutchouc.

In addition to the very numerous inventions relating to the polymerisation of isoprene, butadiene, and other unsaturated hydrocarbons and the coagulation of the emulsions produced, there are many others relating to the manufacture of rubber substitutes from more complex starting materials. Thus, to mention only a few, there are processes for heating fatty oils with various organic and inorganic modifying agents, and vulcanising the modified material (321,724-6, Vol. XXII, 57), processes for oxidising paraffin hydrocarbons or waxes and heating the products with glycerol or other polyhydric alcohols (322,496, Vol. XXII, 125; 327,094, Vol. XXII, 519), and processes for the polymerisation of mineral oils free from constituents boiling below 205° C. (333,901, Vol. XXIII, 392.)

### Metallurgy

In the metallurgical field it is at once apparent that there has been for some time past a shifting of interest from the processes for the extraction of the metals from their ores to the processes for improving the qualities of the extracted

metals to adapt them to innumerable purposes both old and new. Wherever a metal or an alloy is used in a machine or an article of commerce it must nowadays have the approved physical characteristics corresponding with the enormous recent advances of knowledge in this field. It is therefore not surprising that most of the recent metallurgical inventions relate to the production, annealing, and refining of every type of alloy, new rust-resisting and acid-resisting steels, titanium steels, silicon steels, and aluminium steels for constructional purposes being especially numerous. Plating processes have been mainly for the electrolytic deposition of chromium, cadmium, or copper, but have included processes for the electrolytic deposition of alloys of predetermined composition, *e.g.*, nickel-iron alloys of predetermined electromagnetic permeability, and the deposition of chromium from an atmosphere containing chromium iodide vapour. The production of metal powders from the metal carbonyls and the production of metal coatings by thermal decomposition of the carbonyls have also been much in evidence. The few inventions relating to the treatment of ores have been mainly concerned with processes such as roasting, sintering, concentrating, and agglomerating, applicable to ores in general, little attention being given to smelting and other processes for the extraction of particular metals.

## The China Clay Industry in 1930

### Disappointment after Promising Start

THE year 1930, which appeared to open with great promise for the china clay industry, has proved to be one of the most disappointing since the War, and it is calculated that the trade of 1930 will show a decrease of nearly £200,000 compared with that for 1929. Nevertheless, since the beginning of December, there has been a considerable revival in the activity of the china clay firms, and there is no doubt that the industry has now reached a high state of efficiency, and is ready to forge ahead with the lifting of industrial depression and recovery in the consuming industries.

There was a big drop in the Board of Trade returns for July, with a corresponding decline in china clay shipments for the same period. The tremendous industrial loss throughout the country was responsible for the shipments of November being not only the lowest among the monthly records for the year, but less than one-half of the volume of tonnage despatched in January.

The two chief channels for china clay are for papermaking and pottery, but the consumption for other subordinate purposes is ever broadening. From the more aluminous clays, sulphate of alumina and of alum are extracted, and it has been extensively employed in the manufacture of ultramarine, especially in Germany. Within the last ten years, through careful and patient research, some firms have established special processes for the purification of the ordinary commercial clays, which are increasingly used for refined purposes.

## An Exhibition Policy

### Advantage of Firms Being Represented by Principals

THE value of principals attending exhibitions at which their firms are displaying was stressed at the recent discussion of the British Chemical Plant Manufacturers on their proposed exhibition in 1931. Dr. Seligman was particularly emphatic about the advantages of meeting rivals and customers and exchanging ideas, and further endorsement of this policy has since been expressed by the Prince of Wales. Referring at the Salesmanship Dinner at Guildhall, London, to the forthcoming British Empire Exhibition at Buenos Aires, where a number of chemical firms are exhibiting, the Prince said:

"I would particularly like, in connection with my approaching visit to South America—to which such kind reference has already been made—to express the earnest hope that the heads of the firms who are exhibiting at Buenos Aires will not only send out first-class men to take charge of their exhibits, but will go out themselves to Argentina while the exhibition is on, see conditions as they are, and establish personal relations with the big buyers out there."

The Prince's recommendation appeals to us with great force, and THE CHEMICAL AGE some time ago decided that it would be represented at the Argentine Exhibition by Mr. John Benn.

## Association of British Chemical Manufacturers

### A Survey of its Activities and those of Related Bodies

*The Association of British Chemical Manufacturers may be regarded as the most central and influential organisation in the industry, and its operations during the past year, as well as those of related bodies, are shortly described in the following article.*

THE Association of British Chemical Manufacturers has had another active year. At the annual general meeting in July, Dr. E. F. Armstrong succeeded Dr. G. C. Clayton as chairman, the latter becoming vice-president. Mr. D. Lloyd Howard is the new vice-chairman, while Mr. E. V. Evans has continued as honorary treasurer. Sir David Milne-Watson has retired from the presidency and has been succeeded by Mr. C. A. Hill, formerly vice-president.

#### Publicity

This year the British Industries Fair was held at Olympia, where the general conditions were much better than at the White City. The Chemical Section was once more organised by the Association. On the whole, the exhibitors were satisfied with the results obtained, though foreign buyers were none too numerous. The system of organised tours for chemical teachers and students, initiated last year, was continued with conspicuous success, and it is confidently anticipated that these visits will eventually redound to the benefit of British manufacturers.

Early in the year, an entirely new edition of the publication entitled *Some British Fine Chemicals* was issued, under the new title of *Directory of British Fine Chemicals produced by Members of the Association*, and widely distributed to potential buyers at home and abroad. The general arrangement of the *Directory* was specially designed to facilitate its use, and in this respect marks a considerable advance on previous issues. The increased number of chemicals listed, as compared with the last edition in 1926, bears testimony to the progress which the fine chemical industry has made, under the protection afforded by the Key Industry Duty on such products.

The Association's main *Directory*, a bi-annual publication, indicating the makers of all chemicals in frequent demand, has been completely revised, and will be issued on January 1, 1931, as the 1931 edition. It will be in six languages as heretofore, and will be of even greater scope than in the past; it will thus constitute an admirable buyers' guide to British chemicals when used in conjunction with the *Directory of British Fine Chemicals*. It will be given a wide distribution throughout the world and should serve as excellent propaganda for British manufacturers.

#### Legislation and Tariffs

There have been no legislative measures directed specifically to the chemical industry during the year, but the Association, through other bodies more directly concerned, has watched the interests of chemical manufacturers in regard to the Coal Mines Bill and the Hours of Industrial Employment Bill, the latter of which will come forward again this Session.

The Association is actively co-operating with the Federation of British Industries in the investigations which the Federation is making, as a result of the overwhelming vote of its members that a change is required in our fiscal policy in order to protect British manufactures, and to develop inter-Empire trade to the full. Data have been supplied to the Federation to show the effect of the Key Industry Duty on the fine chemical industry, while a detailed investigation into the imports of chemicals into the Empire has been made by the Association as a preliminary to some concerted plan of campaign to secure a larger share of this trade. The chemical import data for 1928, the last year for which detailed figures were available at the time of the inquiry, show that there are some £14,000,000 worth of chemicals, drugs, dyes and colours imported into this country, of which a large proportion can or could be made here; as regards the rest of the Empire, this country only secures 38 per cent. of the import trade, leaving a balance of £23,000,000 worth of chemicals which come from foreign countries but which could well be supplied from the United Kingdom. It is hoped that this investigation will be productive of useful results and will lead to the increased development of our chemical industry, particularly if backed by a suitable fiscal policy.

#### Dyestuffs (Import Regulation) Act, 1920

The question of most vital interest to the industry has been the future of the Dyestuffs (Import Regulation) Act, 1920, which expires on January 14, 1931. The Dyestuffs Industry

Development Committee of the Board of Trade, on which both users and makers are represented, submitted a report in July setting forth a collection of agreed facts and figures regarding the operation of the Act. This report, which made no definite recommendation in regard to future legislation, was published early in September, and was followed immediately by a memorandum from the Colour Users' Association in which it was urged that as the Act was for 10 years and no longer, it should be allowed to lapse. The same week, at the British Association meeting at Bristol, there was a discussion regarding the Act and the benefits which had accrued from it to British industry and research.

Of special note was the contribution made by Mr. James Morton under the title "Dyes and Textiles in Great Britain, 1930," in which he reviewed the operation of the Act as a colour user, and brought forward facts and figures—which no one has been able to refute—to prove that the Act has rendered a great service to the dye users in this country, and that the objections which have been levelled against it by certain colour consumers will not bear critical examination. This contribution was printed in pamphlet form and widely distributed by the Association to all Members of Parliament, leaders of science and industry and the Press, as was also the memorandum issued shortly afterwards by the Association, setting forth the views of the chemical industry. The issue of this latter document was withheld while negotiations were in progress between representative makers and users under the aegis of the Board of Trade, but as soon as these broke down, publication became necessary to ensure that the views of the dyemakers received the same prominence as those of the users.

The Association's Memorandum urged that a further period of protection be given to permit of the completion of the work which it was the object of the Act to achieve and which had already made such good progress. In order, however, to remove the last trace of economic handicap to the users, the dyemakers expressed their willingness that prohibition to import should only apply so long as the British maker was willing to supply an equivalent product at an equal price, provided that it was not a dumping price. This would permit of the continued development of this branch of the organic chemical industry, with all the numerous advantages which accrue therefrom in many directions, without imposing any economic hardship on the user, since the latter would never be called upon to pay any price higher than his foreign competitors. The users, however, did not accept this offer. Strong support of the makers' case has been received from many quarters; the Institute of Chemistry has addressed a Memorandum to the Government from the point of view of the employment of chemists, while Dr. E. F. Armstrong and Sir William Pope have addressed the Society of Dyers and Colourists and the British Science Guild respectively in support of an extension of protection.

In spite of every representation the Government decided that the Act should lapse on the appointed day, that is January 14, 1931. This was followed by an active correspondence in the Press, and was the subject of a debate in the House of Commons on December 4; after a most interesting discussion, the Conservative amendment, that the Act be continued for a further period of five years under the Expiring Laws (Continuance) Act was lost by only 30 votes. The Government's main arguments in support of this decision were that the industry had made such remarkable progress and was now so organised that the danger of intensive foreign competition, which was urged from many quarters, was not really serious, and that the incidence of the Act on the textile industries, with its alleged restriction on the supply of dyes, had been an important factor in producing the present depression and must be removed if these industries were to improve their position. While the dyestuffs industry could make every effort to restrict the inroads of foreign competition into an unprotected British market, it has powerful forces arrayed against it. At once the effects of the Government

decision were felt; new developments were suspended; men were discharged, while the more dangerous foreign competitors made arrangements to send large quantities of dyes into this country as soon as the Act lapsed.

The chemical industry feels strongly on the following three points:—

(1) That the Government decision was unduly delayed and did not give the industry adequate time (it was less than two months) to make arrangements to cope with the adverse conditions under which it will have to be conducted.

(2) That while all the facts in support of the makers were made public for anyone to criticise and challenge, the data on which certain sections of the users, and in particular the textile trade, have based their objections, have not even yet been published. Thus the industry has not only been given no opportunity of replying to the criticisms, but is in fact still unaware as to the precise arguments advanced.

(3) That such a novel and important experiment in political economy as the Dyestuffs Act has not been made the subject of an impartial investigation on judicial and scientific lines, removed from the area of party politics, so that the country at large may be given an authoritative statement of all the facts of the case.

It is felt that in a matter of such national importance the Government decision should not have been taken until there had been a full inquiry into all the facts, and this the chemical industry would welcome. As a result of action taken by the House of Lords, the Dyestuffs Act has now been extended for one year under the Expiring Laws Continuance Bill.

#### Safety in the Chemical Industry

The Association, through its Works Technical Committee, has continued its activities in connection with safety in chemical works. Good progress has been made with the preparation of Part II of the Model Safety Rules which contains a detailed explanation of the Rules in Part I with useful suggestions as to methods of application. The General Section (Section I) and Sections II and VI, dealing with the design and operation of plant with fire and explosive risks, have been completed in draft, and have been issued in provisional form to all members of the Association for their criticisms.

The Safety Circular service initiated last year has been continued, and to date some 40 circulars, dealing with accidents of an unusual nature or from which a useful lesson may be learnt, have been issued to members of the Association and its affiliated bodies, the normal distribution being 1,250 copies. Many firms have written to express their appreciation of these circulars and of their value in suggesting safeguards.

The Association has also decided to issue a quarterly Safety Summary as a regular feature. This Summary will contain reports of accidents which are not of sufficient value to justify issue as safety circulars, and abstracts from the published literature of information bearing on safety in the industry. It is hoped in this way to bring together in a concise form for the use of members all material likely to be of assistance in conducting a safety campaign. This is bound to lead to a diminution in the number of accidents, with reduced losses arising from injury to personnel and damage to property. The Summary has been welcomed by the members, and some 1,200 copies are at present being distributed, though the scheme has been in operation for only a few months.

The safety work of the Association is being done in close liaison with other interested bodies in this country, such as the Home Office, the Chemical and Allied Employers' Federation, the National Safety First Association, etc., while a scheme of collaboration has been arranged with the French and German chemical industries.

#### Standardisation

The Association has actively taken up a proposal that a British Chemical Standardising body should be constituted which would do for chemistry what the British Engineering Standards Association has done and is doing so well for engineering. A conference of all the chemical interests was held in June. Enthusiastic support was received for the idea, and it was unanimously decided that a chemical standardising body should be set up, but that in the interests of efficiency and economy it was essential that there should be only one standardising organisation in this country, which should embrace all forms of standardisation. An *ad hoc* committee was forthwith appointed to explore the situation in collaboration with

the British Engineering Standards Association, which had indicated its willingness to enlarge its organisation so as to include all forms of standardisation. This committee has been at work and negotiations with the British Engineering Standards Association are now in train. It is confidently hoped that in the near future a scheme of organisation suitable to all parties will be developed, so that chemistry and engineering may work on an equal footing in the new standardising body.

#### Miscellaneous

The most noteworthy feature of the year's work is the rapid growth of the co-operative spirit, which is breaking down the old barriers between manufacturers, between different sections of industry and between the various Societies, Associations, and Institutions connected with chemistry and its applications. This is a splendid augury for the future for, with real co-operation, there are few obstacles to progress which cannot be overcome.

#### British Chemical Plant Manufacturers' Association

The British Chemical Plant Manufacturers' Association has had another active year and can record a further increase in membership. At the annual general meeting in July, Mr. J. Arthur Reavell, of Kestner Evaporator and Engineering Co., Ltd., retired from the post of chairman which he had held for the past three years, and was succeeded by Dr. H. J. Bush, of Huntington, Heberlein and Co., Ltd. Mr. N. E. Rambush, of Ashmore, Benson, Pease and Co., Ltd., was elected vice-chairman, while Mr. E. A. Alliott, of Manlove, Alliott and Co., Ltd., was re-appointed honorary treasurer.

The Association has continued its work of publicity and propaganda on behalf of British chemical plant, and has directed its attention primarily to the Canadian and South African markets. It has also revised its official directory, now a recognised buyers' guide for all types of plant for the chemical and allied industries, and a new edition of extended scope and utility will appear at the beginning of January as the 1931 edition to replace the 1929 issue. This Directory will be widely distributed throughout the world, as in the past, while copies may be obtained from the Association's office at 166, Piccadilly, free of charge on application.

Attention has been drawn in the technical press to the striking display of chemical plant and apparatus at the German Achema at Frankfurt in June, and suggestions were made that this example might be followed with advantage by British makers of chemical plant and equipment. The jubilee celebrations of the Society of Chemical Industry next July will afford a unique opportunity for bringing the products of the British chemical plant industry to the notice of interested industrialists at home and abroad, and steps are therefore being taken by the Association to organise an Exhibition of British plant, apparatus and constructional materials used by the chemical industry, interpreted in its widest sense. The Exhibition will be at Central Hall, Westminster, from July 13-18 inclusive. Any maker of British plant and apparatus will be allowed to exhibit even if he is not a member of the Association. It is confidently hoped that the Exhibition will receive the full support of the industry and that it will be possible to stage a display which will be as attractive as the Achema and which will afford a convincing demonstration of the ability of British makers to provide the most up-to-date and reliable plant in the world. In order to stimulate interest in the proposal, the Association arranged at its annual dinner in November for an after-dinner discussion on the question of whether chemical plant exhibitions are of any use. This discussion, in which both users and makers of chemical plant took part, showed conclusively that well-organised exhibitions can be of the greatest utility in promoting trade.

The Monthly Summary of Information on Chemical Engineering, which was started last year, has now become a regular feature of the Association's work and is proving of great value to members. The possibility of extending its sphere of usefulness by co-operation with other bodies has been discussed but, owing to difficulties, mainly of a financial nature, little progress has been made.

The Association has been engaged, in conjunction with the Association of British Chemical Manufacturers, in the drafting

of a standard set of contract conditions for the purchase of chemical plant, and the work has made good progress. The two Associations have also co-operated in the investigation of the whole field of chemical plant standardisation, and have submitted a report to the British Engineering Standards Association in which definite suggestions are made as to the lines along which further standardisation might be profitably considered.

### The Association of Tar Distillers

The main activities of the Association of Tar Distillers during the past year have been concerned with the problem of finding new outlets and uses for creosote. The Creosote Propaganda Committee, to which reference was made last year, has broadened the scope of its activities, and, as an indication of its wider functions, has changed its name to that of the National Creosote Committee. Its early investigations into the preservation of timber indicated that a great deal of educational and propaganda work was necessary to ensure a better utilisation of the wood used for constructional and other purposes, and to this end the Committee have been instrumental in bringing about the formation of the British Wood Preserving Association, which came into being in the early part of the year. This Association is an entirely separate body on which all the interests concerned, including those of growers, suppliers and users of timber, are represented. It is making excellent progress and doing valuable work in spreading information in regard to timber preservation from all aspects.

The National Creosote Committee has continued its investigations in the foreign markets, but, as such developments are likely to be slow, attention has been concentrated on the possible utilisation of creosote as a fuel for Diesel and low-pressure internal combustion engines. There are promising indications in these directions, but much requires to be done to overcome the various chemical and engineering difficulties which are involved. The various developments in regard to the hydrogenation and cracking of creosote oil have also been watched.

The Association of Tar Distillers has made a survey of the position in regard to research on tar, its distillation and its distillation products, and definite proposals have been formulated which will receive due consideration when the time is ripe.

The subject of pitch warts has been kept under close review from every angle, and the Association has been gratified by the fact that the instructions which it prepared for its members in regard to regular medical examinations have found ready acceptance by firms outside the Association.

The Association has collaborated with the Association of British Chemical Manufacturers in the work which is being done on safety in chemical factories and in other problems of common interest.

On completion of his year in the chair, Mr. J. H. Olliver, of the Gas Light and Coke Co., was succeeded by Mr. F. C. Tudsberry, of Scottish Tar Distillers, Ltd., while Major A. G. Saunders, of Burt, Boulton and Haywood, Ltd., has continued to act as honorary treasurer.

### British Disinfectant Manufacturers' Association

The new British Disinfectant Manufacturers' Association, the early formation of which was foreshadowed last year, was inaugurated in December, 1930. It has received enthusiastic support from the trade, and the present membership of 55 represents well over 90 per cent. of the disinfectant production of this country. The chairman of the Association is Mr. N. F. Kingzett, of Sanitas, the vice-chairman is Mr. W. H. Hivey, of Taylor's Automatic Disinfectant, Ltd., and Mr. R. A. Blair, of Burt, Boulton and Haywood, Ltd., is honorary treasurer. The secretary is Mr. J. Davidson Pratt, the general manager of the Association of British Chemical Manufacturers, to which the new organisation is affiliated. The British Disinfectant Manufacturers' Association has in view the normal objects of a trade association, but it has expressly excluded by its rules all questions of price control or regulation. It is imperial in its nature, and any individual firm or company manufacturing disinfectants in the British Empire is eligible for membership.

The first task which the Association had to undertake was to formulate a standard method for comparing the bactericidal value of disinfectants. After a careful and critical review of all existing practices, it adopted the principles of the Rideal Walker method. The technique of this test has been revised and standardised in such a manner as to ensure concordant results in the hands of different workers. Discussions are now proceeding with a representative Government Committee which is considering the same subject, and on which there are strong advocates of the Martin Chick test as representing more nearly practical conditions than the Rideal Walker method. The British Disinfectant Manufacturers' Association is strongly opposed to the Martin Chick procedure, since this test, in its present form, does not satisfy the requirements of a standard test in regard to either the use of readily available standardised materials or the concordancy of the results in different laboratories.

Difficulties have arisen in regard to the use of lysol for midwifery purposes, owing to the alleged unsatisfactory nature of certain lysols and lysol solutions on the market. It is important that a suitable standard for lysol should be set up in order to remove these objections, and the Lysol Makers' Section of the Association is now actively co-operating with the Pharmaceutical Society and the Pharmacopoeia Commission in the preparation of a standard specification.

The Association has also submitted its views to the Government on the recent report of the Departmental Committee on the Poisons and Pharmacy Acts, and it has advised the Governments of various parts of the Empire in regard to the revision of rules and regulations pertaining to disinfectants and their use.

### Another Year for Dyestuffs Act

#### Submission of the Government

THE Dyestuffs (Import Regulation) Act, 1920, received a further lease of life on Thursday, December 18, when the House of Commons decided, on the motion of Mr. W. Graham, not to insist on its disagreement with the Lords' amendment to the Expiring Laws Continuance Bill to prolong the Act until December, 1931. Earlier in the day the House of Lords had insisted on its own amendment, and the sudden and somewhat unexpected submission of the Government was the cause of considerable excitement in the Commons. The name of Imperial Chemical Industries was frequently mentioned during the debate.

Mr. Henry Mond, in the course of a reply, said that charges or imputations had been made against himself and other directors of the company with which he was connected. He had made no secret when he had addressed the House of his connection with that company. He could not have made plainer how little he or those associated with him desired any stringent measure of protection or any unfair advantage. He had been in a position to speak for half of the dye-making industry, and had said that he was prepared to accept one year's continuation with a judicial inquiry in place of the five years put forward by the Conservative party.

The stages in the Dyestuffs Act fight in Parliament have been as follows:—

- December 4.—Conservative amendment to include the Act in the Expiring Laws Continuance Bill and prolong it for five years defeated in House of Commons by 30 votes.
- December 15.—House of Lords, by 73 votes, includes amendment to prolong Act by one year.
- December 17.—Government motion to disagree with Lords' amendment carried by 6 votes.
- December 18.—Lords insist on their amendment. Commons accept amendment.

### Propionic Acid and Safeguarding Duty

A REPRESENTATION regarding propionic acid has been made to the Board of Trade under Section 10 (5) of the Finance Act, 1926, which allows the Treasury to exempt from Safeguarding duty any article which they are satisfied is not made in sufficient quantities in any part of His Majesty's Dominions, having regard to consumption in this country. Any communication should be addressed to the Principal Assistant Secretary, Industries and Manufactures Department, Board of Trade, Great George Street, London, within one month.

## Chemical and Allied Societies : Their work during the Year

*The following specially contributed notes on the work of the principal chemical and allied societies indicate both the range and the importance of their work, and record many interesting developments during the year.*

### British Association of Chemists

There has been a further substantial increase in the association's membership during the past year, the total membership having increased during the past five years by nearly 85 per cent. This, at a time when the membership of most societies has tended either to remain stationary or to fall, is regarded as very satisfactory. Owing to the increasing activities of the association, the office accommodation has been enlarged, but the headquarters of the society remain at Empire House, Piccadilly.

The financial position of the association both in regard to the General Fund and the Unemployment Benefit Fund is satisfactory. Owing to the increased membership the number of units taken up has enormously increased. As a result of this increase and the consolidation of the reserve fund the Council, upon the recommendation of the Unemployment Special Purposes Committee, has increased the rate of benefit by a restoration of that in force prior to 1926. The calls upon the fund have been heavy as a result of the depressed conditions of industry, but despite this fact the percentage of members unemployed has remained low. This is largely due to the increasing efficiency of the Appointments Bureau, which is rapidly becoming one of the most important of the association's activities. The Council looks forward to the time when the lists issued weekly by the Bureau will consist almost entirely of posts negotiated directly by the association. Hundreds of employers have approached the association with a view to obtaining suitable applicants for positions in industry, and in the large majority of cases it has been possible to supply a suitable candidate. The rate of salary offered has been satisfactory. The average minimum salary has been £400 per annum and in many cases, of course, much higher. The Bureau has been particularly successful in filling highly specialised, and therefore highly paid, appointments, and in many cases has succeeded in supplying a suitable candidate where extensive advertisement has failed. The Bureau is, of course, in close touch with the Ministry of Labour, and has been able to supply British chemists in cases where it was first thought that foreign practitioners were essential for the particular post in question. Chemists requiring, and able to fill, highly paid appointments are recommended to get in touch with the association.

The Legal Aid Department has been more active during the past year than in any since its inception. A very large number of cases have been dealt with relating to the matter of inadequate notice, and in all cases members have received the equivalent of at least three months' notice and in some cases six months. In only one case of this kind has it been necessary to proceed to litigation. In this decision not only did the member receive damages in lieu of proper notice, but his professional competency, called into question by the defendants, was vindicated by the judge in unequivocal terms. In a still more important case a member received £1,400 in respect of bonus claimed by him by reason of metallurgical improvements. Points were raised in this case which made it one of the most important of its kind in recent years.

In the matter of the organisation of the profession the Registration Committee of the Council has forwarded a letter to all societies likely to be interested regarding the formation of a General Chemical Council. The newly constituted Federal Council for Chemistry appears to be a body which may be able to fulfil functions of this kind, and the association hope that in the near future some definite move will be made either by this body or some other to put into practice suggestions which the association has consistently advanced for more than ten years.

The association views with regret the proposed operation of a scheme to house in one building a number of scientific societies irrespective of the branch of science these societies represent, as being one which—however admirable from a practical point of view—will do little or nothing to bring

together the societies which represent chemists. As it did in 1926 the association still advocates the principle of a chemistry house, a principle which cannot be applied in practice, even in the distant future, should the present scheme materialise.

Both in regard to the General Chemical Council and any plan for a communal building, the association again emphatically asserts that professional organisation is impossible which does not rest upon a basis of close understanding and co-operation between societies which represent chemistry and chemical industry. Co-operation with other scientific societies, however desirable, can accomplish nothing until this indispensable condition is fulfilled.

The meetings and social functions of the association have been increasingly well attended during the past year. Evidence is not lacking that the members of the association are, in the matter of policy, particularly interested in the question of organisation of the profession and registration. The Council is unwilling to proceed with its programme in this connection without the co-operation of other societies, since it has always held, and still remains of the opinion, that the work of registration ought not to be the work of any one society alone; but the Council, with the consent of its members, will not hesitate to proceed if for any reason this co-operation cannot be obtained in the near future.

### British Engineering Standards Association

Among the work which is being carried out by the British Engineering Standards Association of direct interest to chemical industry is the preparation of a series of British Standard Specifications for Gas Cylinders. British Standard Specification No. 399, High Carbon Steel Cylinders for the Storage and Transport of Permanent Gases, was issued in October of this year, and two further specifications dealing with Low Carbon Gas Cylinders for Permanent Gases and Gas Cylinders for Liquifiable Gases are shortly to be issued. These specifications have all been based on the recommendations included in the Reports of the Gas Cylinder Research Committee of the Department of Scientific and Industrial Research. At the same time specifications are nearing completion for Valves for Gas Cylinders and for Identification Colours for Gas Cylinders. The latter specification provides for Identification Colours for gas cylinders for the gases most commonly in use the underlying principle of the scheme being that yellow should represent toxic gases and red inflammable gases.

Consideration is now to be given to the question of Welded Containers.

It is hoped that the British Standard Specification for Testing Sieves will be published early in the new year and in order to assist the committee in the further work of preparing a British Standard Schedule of Sieves and Screens for industrial purposes a questionnaire is being widely circulated to obtain the fullest possible information as to current practice.

It may be of interest to mention that, in accordance with the procedure by which British Standard Specifications are periodically reviewed so as to bring them up to date, the following B.S. Specifications are now being reconsidered by the Committee responsible for their preparation: B.S.S. No. 185, Benzol for Motor Fuel; B.S.S. No. 186, Steam Jacketed Pans; B.S.S. No. 334, Chemical Lead.

In the Aircraft Section a certain amount of work of direct interest has been carried out in the revision of the series of Dope and Dope Ingredient Specifications issued during the past year. Not least amongst these is Specification 3.D.15, "Distillation Apparatus."

The list of B.S. Specifications for Non-Ferrous Metals has been appreciably increased this year by the addition of B.S. Specifications for Gun Metal, Phosphor Bronze, Cupro-Nickel and Aluminium, and Aluminium Alloys, and all these find a place in various phases of chemical engineering.

### Chemical Society

During the year under review the Chemical Society has continued steadily to pursue the objects for which it was founded nearly 90 years ago. Fifteen ordinary scientific meetings have been held, at which 62 papers were read and discussed, and up to the end of November, the Journal contains 340 papers, occupying 2,582 pages compared with the corresponding figures of 355 and 2,661 respectively for the previous year. The number of pages in the Abstracts "A" dealing with Pure Chemistry to the end of November, 1930, was 1,486, as against 1,348 at the same period in 1929. The Annual Reports for 1929, Volume XXVI (published in February, 1930), contained an addition to the usual reports, a biennial report on Mineralogical Chemistry for the year 1928-1929, and a report on Crystallography. Volume XXVII for 1930 will contain a special report of Conductivity and a biennial report on Radioactivity and Sub-Atomic Phenomena.

The Faraday Lecture, entitled "Chemistry and the Quantum Theory," was delivered by Professor Niels Bohr, on May 8, and will be published in the Journal and also in separate form. The Liversidge Lecture, entitled "Fifty Years' Experimental Research upon the Influence of Steam on the Combustion of Carbonic Oxide (1880-1930)," was delivered by Professor W. A. Bone on December 11th, and arrangements have been made for Professor H. Wieland to deliver the Second Pedler Lecture, entitled "Studies on Biological Oxidation," on March 6, 1931.

A special meeting of the Society was held on October 16, when a plaque of the late Professor W. H. Perkin, F.R.S., presented by subscribers to the Perkin Memorial Fund, was unveiled and an oration on the life and work of Professor Perkin delivered by Professor W. N. Haworth.

Applications for grants for research work amounting to £929 10s. have been received during the year, and grants amounting to £665 have been made. Owing to the fact that the sum applied for each year far exceeds the amount available for distribution, prior claim has to be given to applications received from Fellows of the Society.

The Library continues to grow steadily in size, the number of volumes, apart from pamphlets, now being over 34,500. It is open daily from 10 a.m. to 9 p.m., except on Saturday, when the closing hour is 5 p.m., and is used by its Fellows and by members of the following contributing societies: Association of British Chemical Manufacturers, Biochemical Society, Faraday Society, Institute of Brewing, Institute of Chemistry, Society of Chemical Industry, Society of Dyers and Colourists, Society of Public Analysts. During 1929, there were 7,567 attendances, and 5,402 books were borrowed.

### Faraday Society

The society has again had a successful year. The material communicated to the Transactions was practically as much as in the record year last year. Two general discussions were held, the first being on "Optical Rotatory Power," and the second was devoted to a colloid subject in accordance with the arrangement which the society made with all the other chemical and physical societies to organise biennial Colloid discussions. The subject chosen was "Colloid Science Applied to Biology"; the plan of campaign was to invite reports from world authorities on special branches of the subject and to circulate these during the long vacation so that the problems of the biologists might be adequately considered by physical chemists. As a result a very largely attended meeting was held at Cambridge at the end of September. The meeting was conspicuous for the number of guests who travelled from all parts of the world to attend it; there were some 25 foreign guests, all of whom contributed materially to the value of the discussion.

The arrangements which were notified last year for an exchange of membership between the American Electrochemical Society, the Deutsche Bunsen-Gesellschaft and the Faraday Society have been working smoothly, and so far as exchange of membership with the Faraday Society is concerned, have now been extended to the Société de Chimie Physique and the Nederlandsche Chemische Vereeniging.

### Institute of Brewing

Commenced in 1920, the Institute of Brewing Research Scheme is financed entirely by the annual subscriptions of corporate members of the Institute. The fund so formed is

administered by the Research Fund Committee for the promotion and assistance of research for the benefit of the fermentation industries generally.

In recent years the Institute has been concerned in researches on barley from two aspects. Firstly to elucidate the influence of soil, season, and manuring on the yield and quality of barley, and secondly from the point of view of its brewing value as determined by the proteins. The results of the field trials carried out in association with the Rothamsted Experimental Station have indicated the value of nitrogenous and other artificial manures and led to a fresh orientation of the advice on manuring of barley given by county agricultural authorities, whereby increase in yield can be obtained without depreciation of quality. The protein work has marked a notable advance in the knowledge of the proteins of cereals and has important applications in brewing. The Research Scheme is also associated with the National Institute of Agricultural Botany in the development of new and improved varieties of barley. A study is being made of the nitrogen requirements of yeast and its application to fermentation. Investigations directed to improvements in the quality of hops are being carried out in association with Wye College and the East Malling Research Station and at the Institute's experimental oast. The active constituents of hops are being studied at the College of Technology, Manchester.

### Institute of Chemistry

Records for the year will show an increase in the roll of members and, compared with the year 1929, an increase in the number of candidates for the examinations of the Institute and for the examinations for National Certificates in Chemistry—the latter being conducted under the auspices of the Institute and the Board of Education jointly. Among the matters which have received the attention of the Council during the past year are: the Royal Commission on Local Government, with special reference to the appointment of Public Analysts and Official Agricultural Analysts; the Poisons and Pharmacy Acts, so far as they relate to professional chemists other than pharmacists, and having special regard to the title of *Chemist*; British Patent Law Reform; Contracts of Service; the Petition for a Royal Charter by the Australian Chemical Institute; and the Dyestuffs (Import Regulation) Act.

An endeavour is being made to secure reliable statistics on the remuneration of professional chemists, the result of which, it is hoped, will be available in 1931.

Representatives of the Council have been appointed to a Joint Committee of the Chemical Society, the Society of Chemical Industry and the Institute, with a view to devising means for promoting closer co-operation among chemical organisations. The position of the Institute in relation to the "Association for the Promotion of Co-operation between Scientific and Technical Societies and Institutions," whose object is to establish a central building for the use of such Societies, appears not to be fully appreciated. The Institute would undoubtedly have been welcomed to participate in the scheme and was invited to do so; but since it already occupies, on definitely economical terms, premises providing over 20,000 square feet of floor area, with facilities for extension and, since such accommodation could not be provided in the proposed scheme on similar advantageous conditions, the Council, while appreciating the thought which prompted the invitation, felt that it was impossible to entertain the proposal.

In addition to many papers of interest and importance which have been read before the Local Sections, a lecture was given in April before the Institute at Headquarters, by Sir Thomas Legge, on "Lessons Learnt from Industrial Gases and Fumes."

The Streiffeld Memorial Lecture was delivered on November 27, by Mr. Bernard F. Howard, Vice-President, who chose for his subject "Some Notes on the Cinchona Industry." The Second S. M. Gluckstein Memorial Lecture was given on December 19, by Dr. A. E. Dunstan, the title of his discourse being "The Transmutation of the Chemist." Sir Thomas Legge's paper has been published separately; a paper on "Food Fakes, Ancient and Modern," by Mr. E. Gabriel Jones, read before the Liverpool and North-Western Section, will be published at the end of the year, and Mr. Howard's lecture on Cinchona is now in the course of preparation for the press. It is hoped that Dr. Dunstan's lecture will also be published in due course.

A new edition of the Register was published during the year, and the seventh edition of the List of Official Chemical Appointments should be ready early in 1931.

The Meldola Medal for 1929 was awarded to Dr. R. A. Morton, and the Sir Edward Frankland Medal and Prize to Mr. Bernard William Bradford. The Pedler Scholarship, which had been awarded to Mr. George Morrison Moir in 1929, was extended until June, 1930, and his work on methods for the estimation of the protein constituents of milk is being published in *The Analyst*. Arrangements for the appointment of his successor will be announced shortly.

From the Sir George Beilby Memorial Fund, administered jointly by representatives of the Institute, the Society of Chemical Industry and the Institute of Metals, awards of £250 each were made in July to Dr. Guy Dunstan Bengough and Mr. Ulick Richardson Evans for their work on corrosion.

Although the demand for the services of chemists has been fairly well maintained, the recent conditions in industry and commerce have somewhat adversely affected the state of employment. During the first half year the number of members of the Institute known to be out of work did not exceed 60, but since July the number has risen to about 100 (rather over 1.7 per cent. of the membership).

There have been greater calls on the Benevolent Fund, but it is satisfactory to record that several members who were without employment and received temporary assistance, have now secured appointments.

#### Institute of Metals

The year 1930 was an important one in the history of the Institute of Metals. It saw a great decision taken by the Council of the Institute to increase the usefulness of its publications. The well-known volumes of the *Journal of the Institute of Metals*, which have hitherto combined both valuable original matter and abstracts, will, commencing from January, 1931, appear half yearly—as heretofore—but without abstracts. The latter will be issued in the form of a separate volume of about 1,000 pages at the end of each year. In addition, members will receive a new monthly issue of the journal containing editorial matter and abstracts that otherwise would appear only half-yearly. The effect of this arrangement will be to speed up the receipt of the abstracts by several months; further, it will make the half-yearly volumes, which had grown rather bulky, much easier to handle. As the new monthly issues of the *Journal* will be available to members only, and will not be purchasable under any circumstances, it is anticipated by the Council of the Institute that a large increase in the Institute's membership will now arise in order that the new publications may be received. The monthly abstracts, it is anticipated, will be cut up for card indexing purposes, as all will appear later in bound form for permanent reference.

During the past year the usual three big annual meetings of the Institute were held. The first was the annual general meeting, which took place in London on March 12 and 13, 1930, when a number of papers were read and discussed; and Dr. Richard Seligman was elected President of the Institute for the years 1930-32. The meeting was followed by a dinner and dance at the Hotel Metropole, which functions were largely attended, not only by British members but also by members from all over the continent of Europe.

In May 1930 the Annual May Lecture was given by Major F. A. Freeth, F.R.S., on "The Influence of Technique on Research."

The final gathering of the year was the annual autumn meeting, which took place at Southampton on September 9-12, when several hundred members assembled to take part in the extensive social programme that had been arranged, and in the reading and discussion of papers. After the discussion a trip was made to France in the *Mauretania*, and with Cherbourg as a centre several hundred miles of enjoyable motoring took place, in the course of which many old cities and pleasant seaside resorts were visited. The return journey from Cherbourg to Southampton was made in the *Aquitania*. The Council was glad to welcome at the Southampton meeting not only many members from Europe and Asia, but also from the United States of America.

Rapid progress has been made during the year, not only in

connection with the visit that the Institute is to pay in September next—13 to 16—to Zürich, but also with the arrangements for the American meeting of 1932. This meeting will be of a combined character, members both of the Institute of Metals and of the Iron and Steel Institute having accepted an invitation from the American Institute of Mining and Metallurgical Engineers to visit the United States in September, 1932. The visit is being eagerly anticipated by the combined memberships of the two institutes throughout the world.

The year 1930 ended successfully for the Institute of Metals both from a financial and numerical standpoint—and this notwithstanding the severe period of trade depression through which the world has been passing. The income surpassed the expenditure actually by a record amount, whilst the membership increased from 2,122 to nearly 2,200.

#### Institution of Chemical Engineers

The continued progress of the Institution is reflected in an increase of membership of about twelve per cent. during the year 1930.

At the annual corporate meeting held in April, Mr. J. Arthur Reavell was re-elected President, and Mr. C. S. Garland and Mr. F. H. Rogers were again elected Vice-presidents. Professor J. W. Hinchley and Mr. F. A. Greene were re-elected respectively honorary secretary and honorary treasurer. The address of the President on "The Rôle of Science in Industry," which aroused great interest in many quarters, gave a comprehensive survey of the many fields in which the application of science gave beneficial results to industry, and included an emphatic plea for a closer and more real partnership between them.

At this meeting the first award was made of the Moulton Medal and of the Junior Moulton Medal, which were established for papers read before the Institution. The Moulton Medal was presented to Mr. H. Hollings, Dr. S. Pexton and Dr. R. Chaplin for a joint paper, and the junior Moulton Medal to Mr. H. Smith. The Osborne Reynolds Medal was presented to Professor J. W. Hinchley.

Two public lectures were delivered during the year: the first in January by Dr. Herbert Levinstein, on "Films and Fibres derived from Cellulose," and the second, in October, by Professor W. A. Bone, on "High-Pressure Gas Reactions." A two-day conference in December on "The Utilisation of Trade Wastes," drew large attendances and evoked lively discussions.

Other papers read during the year included, at the annual corporate meeting, "Pulverised Fuel," by Dr. J. T. Dunn and Dr. Burrows Moore, and "The High-Pressure Equipment of the Chemical Research Laboratory, Teddington," by H. Tongue; and in October "The Effect of Surface Conditions on Heat Transmission," by Dr. S. J. Kohli.

Six meetings were held by the Graduates and Students Section during the year, among which the paper by Mr. O. A. Saunders on "Heat Transfer Calculations" was especially noteworthy.

The President's Reception, held on November 12 at the New Burlington Galleries, London, again attracted a large and distinguished gathering, some five hundred members and friends being received by Mr. Reavell and his daughter, Mrs. J. F. Attenborough.

The Associate-Membership examination of the year followed the lines of that in 1929, and the report of the Board of Examiners commented upon the improvement in the knowledge on fundamental questions and indications that the requirements of the Institution were becoming better known.

#### The National Benzole Association (N.B.A., Ltd.)

During the past year the Joint Research Committee of the National Benzole Association has made further progress in a number of problems which have previously engaged the attention of the Committee. The possibility of preventing during storage the resinification of comparatively crude benzenes by the addition of inhibitors, and of subsequently utilising such benzenes satisfactorily as motor fuel, has now been more thoroughly tested on the technical scale. Fleet trials carried out by the Gas Light and Coke Co. have demon-

strated that stabilised gas works benzole, produced under normal working conditions, gives satisfactory results, no gumming of engines having been observed. Further trials on coke-oven benzoles are now in progress. Owing chiefly to the discovery that certain types of cracked petrols possess appreciably higher anti-knock values than straight run petrols, the work of the Committee on the problem of the gumming of motor spirits has recently attracted considerable attention.

Further investigations have been made on wash oils for benzole recovery, such as an examination of the absorptive capacities and stabilities of sulphur treated creosotes. The deterioration of creosote oil and gas oil when used under working conditions for the recovery of benzole from coke-oven gas has also been carefully investigated, and much information obtained of value to benzole producers. Owing to differences in the design and operating conditions of the plants, however, no comparison was possible from the point of view of costs and efficiencies of recovery with these two oils.

Owing to the growth in the research activities of the committee, the research staff of the National Benzole Association has now been transferred to a new laboratory at Willesden, and the connection of the Association with the University of Leeds has been terminated. The work previously carried out by the Joint Benzole Research Committee is now being continued by a Committee composed of members of The National Benzole Association.

#### Society of Dyers and Colourists

Meetings and lectures under the various sections of the society have been held in Bradford, Huddersfield, Manchester, London, Leicester, Nottingham, Derby and Glasgow, particulars of which are contained in the January and November Journals of the society.

The Gold Medal of the Worshipful Company of Dyers was awarded in the year 1930 to: Mr. F. Scholefield, M.Sc., F.I.C., for his paper "The Action of Light on Dyed Colours."

The Gold Medal of the Society of Dyers and Colourists was also awarded in 1930 to Mr. Arthur Silverwood, for "Exceptional Services rendered to the Society as Honorary Secretary 1913-1930."

In connection with the "Colour Index" of the Society the demand for this work still continues, and also for the "Supplement" to the Index published in 1928.

The work on "Standardising the Methods of Testing the Fastness of Dyed and Coloured Materials" is progressing very favourably, and during the present year a number of Interim Reports have been published.

#### Society of Glass Technology

During the year the society held nine meetings: four in Sheffield, one in London, and one each at Leeds, Stourbridge, Buxton and Stoke-on-Trent. At these meetings twenty-eight papers were communicated. In addition, there were five meetings of the London Section of the society.

Two events were of very special interest during the year. The first was a joint international meeting which brought to England from May 26 to June 3 a party of some sixty members of the Deutsche Glastechnische Gesellschaft. A series of visits to glass works in different parts of the country was arranged and joint meetings were held in London. The Technical Committees of the two societies also met in London to discuss matters of general interest and the formulation of standard tests and specifications. The second important event in which the society was actively interested, along with other bodies, was the holding at Buxton from September 17 to 20 of the Second Glass Convention under the Presidency of Mr. Walter Chance.

Early in the year the society published its second brochure, namely "Notes on the Analysis of Glasses, Refractory Materials, and Silicate Slags." Visits of the society have been made to seven industrial plants, as well as to other institutions.

At the annual general meeting Mr. Christopher Wilson was elected President for the year 1930-31. The general treasurer is Mr. Joseph Connolly, Salford; the American treasurer, Mr. F. C. Flint, Washington, Pa.; and the secretary, Professor W. E. S. Turner, of Sheffield.

#### Scottish Coal Tar Products In 1930

(By Our Scottish Correspondent.)

WHILE trading at the beginning of the year was quite satisfactory from almost every point of view, it is unfortunately the case that the second half of the year has more than wiped out any advantages which were gained during the first six months. The price level during January/June was sufficiently high to give the distillers a reasonable return on their products, and it cannot be said that consumers were unwilling to pay the prices being asked. From July onwards, however, it became apparent that production was much greater than the demand and, consequently, prices came tumbling down with a rapidity unequalled since the big slump following the General Strike of 1926. Consumers who had contracted for their estimated requirements found themselves unable to take up their contract quantities and distillers, with storage capacity already taxed very severely, were left without any outlet for the bulk of their production. The cause of this sudden depression has never been explained satisfactorily, the only reason advanced being the falling away of the world demand for all commodities. So far as this country is concerned the depression in the export side of the by-products business is partially, if not wholly, caused by the ever increasing production, synthetically and otherwise, of the self-same products in countries which, in the past, were our best customers. It is considered, however, that when the present depression (which is not confined to tar products) is over, the world demand will be sufficient to take care of the ever increasing production.

In last year's report it was mentioned that steps were being taken to find new uses for creosote oil, but very little headway has been made in this direction.

No change is anticipated during the early months of the coming year, but already there are signs which appear to indicate that values are at their lowest and that the turning point has been reached. Some large consumers are taking advantage of the present low prices to cover themselves for forward delivery. It should be remembered, however, that the potential supply of coal tar by-products is large, and therefore, rapid price fluctuations are unlikely to take place.

#### Prices during the Year

*Cresylic Acid.*—The demand during the first few months of the year was good, and the value of pale 97/99% quality was not below 1s. 10d. to 1s. 10½d. per gallon. From July onwards, however, quotations fell rapidly to the present level of 1s. 6d. to 1s. 6½d. per gallon, and even at this low figure it is difficult to find an outlet for available supplies. Nevertheless a note of optimism can be struck when it is considered that there is an ever increasing production of synthetic resins, some of which are made from tar acids.

*Carbolic Sixties.*—There was an excellent demand during January/June with value steady at from 2s. 4d. to 2s. 6d. per gallon for grades containing over 5% water. Thereafter orders dwindled and value fell until to-day there is no market even at the extremely low prices now ruling, namely, 1s. 6d. to 1s. 8d. per gallon for the same qualities. Grades containing a low water content were selling at 2s. 10d. to 3s. per gallon during the first few months and now stand at the nominal price of 1s. 10d. per gallon.

*Creosote Oil.*—The demand has been on very modest lines during the year and, taking Specification oil as standard, value has fallen from round 4d. per gallon in January to 2½d. per gallon at the present time. Virgin oils have found a regular, if small, outlet throughout the year and the price level has remained steady at 3d. to 3½d. per gallon.

*Coal Tar Pitch.*—The demand for Scotch pitch has been poor all year and, although efforts were made to form a control in Scotland, the proposition did not meet with any marked success. Few shipments have been effected and price has fallen from round 50s. per ton in January to 42s. 6d. per ton in December.

*Blast Furnace Pitch.*—The controlled prices have remained stationary all year. The volume of business has been very disappointing, but, as production was curtailed very considerably owing to the ironmasters reducing the number of furnaces in blast, stocks have been kept within reasonable limits. The controlled prices were, and still are, 30s. per ton f.o.r. works for home trade and 35s. per ton f.a.s. Glasgow for export.

(Continued on page 630).

## A United Effort for a Chemical Headquarters

From a Correspondent

THE year has witnessed the fusion of two movements neither of which would in all probability have been possible of realisation by itself. Both movements started about the same time, the one having for its object the housing under one roof of the chief mining and metallurgical societies and institutions of the Empire, and the other aiming at the erection of a common building to house the chief societies and institutions connected with chemistry.

The two movements were entirely separate and distinct. The preliminary step in connection with the mining group was taken in 1921, when the Institution of Mining and Metallurgy (representing the mining of minerals other than coal, and the production of metals other than iron and steel), entered into an arrangement for closer co-operation, with a combined secretariat, library service, etc., with the Institution of Mining Engineers (representing coal-mining engineering). The next step was taken in 1924, when, at the First Empire Mining and Metallurgical Congress, held in connection with the British Empire Exhibition, the Empire Council of Mining and Metallurgical Institutions was formed, the chief object of which was to serve as an organ of inter-communication and co-operation between the constituent bodies, and to promote their common interests. A further step was taken in August, 1927, when, at a meeting of the Empire Council held at Ottawa, a resolution was passed emphasising the importance of securing a building in London as a permanent centre for the Empire Council, its constituent bodies and allied institutions, and requesting Sir Thomas Holland (chairman), Sir John Cadman (treasurer) and Mr. C. McDermid (hon. secretary) to consider the question and formulate proposals.

### Chemistry House

So far as the chemistry group is concerned, the idea of a "Chemistry House," containing the chief chemical societies with a central library, club, and meeting room, has engaged the attention of chemists for many years past. The first scheme to assume any definite form was the one which, shortly after the war, enlisted the interest of Lord Moulton, and it is certain that this scheme would have come to fruition had not the untimely death of that eminent man and great organiser led to its abandonment in the initial stages. Subsequently other schemes were suggested, but each failed to convince people of its soundness, mainly because the collection of the large sum of money deemed necessary seemed totally beyond the capacity of the science and industry of chemistry.

Meanwhile the need was becoming increasingly urgent. The Chemical Society had long outgrown its premises in Burlington House, and was rapidly reaching the stage when no more books could be accommodated in its library. The Society of Chemical Industry and the Institution of Chemical Engineers had only office accommodation, and relied on the Chemical Society for the use of its meeting room—an apartment totally inadequate for the purposes required, being much too small and singularly badly ventilated.

When, therefore, the exploring committee, which had been formed by the mining group, in 1928, approached the members of the chemical group in June, 1929, and suggested co-operation in bringing their scheme to a successful issue, it was at once apparent that what was not practicable for one group alone might be easily feasible if a combined effort was made by both groups acting together. A basis of agreement was soon reached, and the Councils of the Chemical Society, the Society of Chemical Industry, the Institution of Chemical Engineers and the Institution of the Rubber Industry agreed to participate in the scheme provided that "the necessary funds were raised and proper accommodation was provided." It was also agreed that the original societies and institutions forming the scheme should be housed at a rental not greater than that they paid for their present premises, and that the Chemical Society—in view of the transference of its library to the new building—should, as in its present apartments, be housed rent free.

### The Leonard Bequest

Before the fusion of the interests mentioned above had taken place—and it must be remembered that it was a true fusion, because it was agreed to pool all resources, unless specific donors wished otherwise—the mining group had

decided to occupy three-fifths of a site in Westminster as sufficient for its needs. When the chemical group joined, it was decided to take over the other two-fifths and assign this section to the group for the purposes of a "Chemistry House." This was not in any way the outcome of a sense of "exclusiveness" on the part of the chemists—as, it is thought, some of our metallurgical friends believe—although, perhaps, sentiment played some part in the desire of chemists to have a "House" similar to that possessed by the Americans, the French and the Germans. The determining factor was indeed a highly practical one, because the munificent bequest of the late Mr. Leonard to the Society of Chemical Industry could only be made available if the trustees were convinced that a project for a definite "Chemistry House" was actually in being early last September. The trustees were satisfied on this point a few days before the claim of the Society of Chemical Industry to the Leonard bequest was due to expire, and the Association was registered on September 15 of this year. It was further agreed that the members of the two groups should be known as "constituent" bodies, namely: (1) *Mining and Metallurgical Group*.—Empire Council of Mining and Metallurgical Institutions; the Institution of Mining and Metallurgy; the Institution of Mining Engineers; the Institution of Petroleum Technologists; the Iron and Steel Institute; the Institute of Metals; the Institute of Fuel. (2) *The Chemistry Group*.—The Chemical Society; the Society of Chemical Industry; the Institution of Chemical Engineers; the Institution of the Rubber Industry.

It was also agreed that the large amount of space in the new building left over after the above-named constituent bodies had acquired adequate accommodation should be leased to "tenant" societies. The rents obtained in this way will, it is estimated, more than cover the cost of upkeep and maintenance of the new building.

As has already been stated, "Chemistry House" will form a definite unit and will comprise a vertical section of the building and have its own entrance. It is proposed to erect a seven-storeyed building, one storey of which—probably the fifth—will be continuous, horizontally, throughout the building and carry the Library—a splendid feature of the scheme—since, to commence with, it will contain some 70,000 volumes, and within a few years will undoubtedly become one of the leading scientific libraries and information bureaux of its kind in the world. It is possible also that the seventh or top floor may be continuous throughout, because it is proposed that this floor should contain the restaurant and club facilities of the building. This will certainly be the case, unless the Chemistry Industry Club wish to become tenants in the building, for, in that event, the top floor of "Chemistry House" (some 3,200 sq. ft.) could be assigned to it, and this floor would be separated, as the remaining five floors will be separated, from the remainder of the building by swing doors. It is understood that the club will consider this question as soon as definite details are available, but should it decide to join, it is evident that the club would retain complete autonomy.

### Forthcoming Appeal

An appeal is being issued to chemical manufacturers for support, because it is upon them that chemists must rely for the greater proportion of the sum of £350,000, which, after careful computation, is regarded as necessary to effect our purpose. An appeal will also be issued to the 20,000 members of the "constituent" bodies, because it is hardly fair to ask for help for the industry, as such, unless the members of the combining organisations are prepared to show that they themselves desire to contribute in accordance with their means.

"Chemistry House" has been allotted two-fifths of the whole site, and chemists must see to it that they provide two-fifths of the sum required. This should not prove impossible, although probably there has been no time worse than the present for issuing an appeal for funds. So far, the response from the industrial side has been very hopeful, and several firms have contributed substantial amounts. But much still remains to be done, although there can be now no doubt that we are within measurable distance of obtaining a "Chemistry House."

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## Patent Literature

The following information is prepared from published Patent Specifications and from the Illustrated Official Journal (Patents) by permission of the Controller to H.M. Stationery Office. Printed copies of full Patent Specifications accepted may be obtained from the Patent Office, 25, Southampton Buildings, London, W.C.2, at 1s. each.

### Abstracts of Accepted Specifications

336,811. ALCOHOLS. H. D. Elkington, London. From Naamlooze Vennootschap de Bataafsche Petroleum Maatschappij, 30, Carel van Bylandtlaan, The Hague. Application date, February 17, 1930.

Ethanol vapour at a temperature about 325° C. and pressure of 130–300 atmospheres is subjected to the action of a catalyst consisting of magnesium oxide containing 20–40 per cent. of copper oxide. Butanol and higher alcohols are obtained.

336,839. LITHOPONE. Sachtleben Akt.-Ges. für Bergbau und Chemische Industrie, Homberg, Niederrhein, Germany. International Convention date, December 5, 1928.

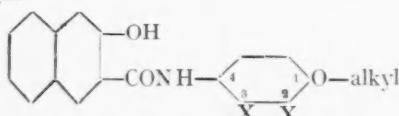
Lithopone is calcined and quenched in water containing magnesium sulphate or sodium sulphate. Barium salts, if not already present, are added to the lithopone before calcination, and the particles of lithopone thus become coated with barium sulphate whereby the lithopone is rendered weather-resistant.

336,922. ALUMINIUM SALTS. Chemische Fabrik vorm. Sandoz, Basle, Switzerland. International Convention date, February 15, 1929.

Aluminium sulphate is treated with alkaline earth gluconates in the presence of alkaline earth hydroxides. The gluconate solution is evaporated to dryness or precipitated with alcohol.

336,938. DYES. W. W. Groves, London. From I.G. Farbenindustrie Akt.-Ges., Frankfort-on-Main, Germany. Application date, July 10, 1929.

A diazo, tetrazo, or diazo azo compound free from sulphonated hydroxy and carboxy groups is coupled with a 2<sup>1</sup>:3<sup>1</sup>-oxynaphthoyl-4-amino-1-alkoxy methyl benzene of the general formula:—



where one X represents hydrogen and the other X represents CH<sub>3</sub>. An example is given in which diazotised 4-chlor-2-toluidine is coupled with the 1-methoxy-2-methyl compound; the dye gives clear red lakes. A large number of additional components and shades are referred to.

336,944. HYDROGEN AND CARBON MONOXIDE. J. Y. Johnson, London. From I.G. Farbenindustrie Akt.-Ges., Frankfort-on-Main, Germany. Application date, April 8, 1929.

Gaseous hydrocarbon mixtures, e.g., containing methane, other than coke oven gases, are introduced with water vapour into a charge in a coke oven, and the gaseous hydrocarbons are converted into hydrogen and carbon or carbon monoxide simultaneously with the carbonisation of coal. An example is given of the treatment of the waste gas from the synthesis of ammonia containing hydrogen, nitrogen, and methane, to obtain a gas containing hydrogen, nitrogen, carbon monoxide and carbon dioxide. Reference is directed by the Comptroller to Specifications Nos. 3,197/1910, 7,049/1910, and 101,152.

336,960. ALIPHATIC ANHYDRIDE. H. Dreyfus, 22, Hanover Square, London. Application date, July 22, 1929.

Vapour of the acid is heated in contact with trioxide or pentoxide of arsenic or antimony, or arsenates or antimonates of sodium, potassium, calcium or magnesium. The catalyst may be contained in a tube of fireclay, silica, or copper, which may also contain filling material. If the catalyst is volatile, a mixture of its vapour with the acid vapour may be passed through a heated tube containing a non-volatile catalyst or filling material. The anhydride is separated from the reaction products in any known manner.

336,970. TREATING CHROMIUM ORES. A. Carpmael, London. From I.G. Farbenindustrie Akt.-Ges., Frankfort-on-Main, Germany. Application date, July 22, 1929.

Chromium ores are subjected to a preliminary heating in an

oxidising gas to over 800° C., with or without a small proportion of sodium nitrate, and are then worked up with alkali carbonate in the presence of lime, dolomite or bauxite.

336,971. DYES. A. Carpmael, London. From I.G. Farbenindustrie Akt.-Ges., Frankfort-on-Main, Germany. Application date, July 22, 1929.

The dyestuffs obtained by coupling tetrazotised 4:4'-diamino-diphenyl-3:3'-dicarboxylic acid with two similar or dissimilar molecular proportions of an amino-naphthol-disulphonic acid or a nuclear substitution product are treated with an agent yielding copper. The resulting o-carboxy-azo dyestuff could also be prepared in the presence of a coppering agent. In an example, the dyestuff benzidine-3:3'-dicarboxylic acid  $\geq$  1:8-amino-naphthol-2:4-disulphonic acid (2 mols.), is treated with copper sulphate. In another example, the tetrazo compound is coupled with H acid in the presence of sodium carbonate, pyridine and copper sulphate. Some dyeing examples are also given.

336,982. DYES AND INTERMEDIATES. W. Smith, J. Thomas, and Scottish Dyes, Ltd., Earl's Road, Grangemouth. Application date, April 16, 1929.

Anthraquinone derivatives containing chlorine, bromine, or sulphonated acid groups in the 2-position, and a halogen atom or diazo group in the 1-position are condensed in the presence of copper. The products are 1:1-dianthraquinonyl derivatives containing the above groups in the 2:2<sup>1</sup>-positions. Examples are given of the treatment of 1:2-dichlor-anthraquinone, 1-amino-2-brom-anthraquinone, and 1-amino-2-sulpho-anthraquinone. The 2:2<sup>1</sup>-substituents in these products may be replaced by other substituents, e.g., by treatment with ammonia.

336,983. DYES AND INTERMEDIATES. W. Smith, L. J. Hooley, J. Thomas and Scottish Dyes, Ltd., Earls Road, Grangemouth. Application date, April 16, 1929.

1:1-dianthraquinonyls containing chloro, bromo, or sulphonated acid groups in the 2:2<sup>1</sup> positions (see 336,982 above) are condensed with substances containing reactive hydrogen atoms, e.g., ammonia, aliphatic amines, aromatic amines, phenols, substances in which the reactive hydrogen atom is attached to sulphur as mercaptans, or alkali salts such as sodium phenate. If ammonia is used, condensation is followed by ring closure of the 2:2<sup>1</sup>-diamino-1:1-dianthraquinonyl to obtain flavanthrone. The 2:2<sup>1</sup>-dichloro derivative may be treated with aniline to obtain a 2:2<sup>1</sup>-dianilino-1:1-dianthraquinonyl, and other examples describe the treatment with methylamine, 1-amino-anthraquinone, phenol, calcium hydroxide and calcium chloride, potassium sulphide.

336,991. DESTRUCTIVE HYDROGENATION. C. F. R. Harrison, Norton Hall, The Green, Norton-on-Tees, and Imperial Industries, Millbank, London. Application date, July 19, 1929.

A metallic catalyst for destructive hydrogenation under pressure in liquid phase is employed in the form of plates, wire, ribbon or gauze. Thus plates may be threaded on rods and spaced by washers to form units which are arranged in a vertical tower with the plates vertical, but arranged at right angles in alternate units.

336,999. PRODUCTION OF ETHYLENE FROM ACETYLENE. J. Y. Johnson, London. From I.G. Farbenindustrie Akt.-Ges., Frankfort-on-Main, Germany. Application date, May 24, 1929.

In the conversion of a mixture of acetylene in hydrogen into ethylene, the catalyst employed is tellurium or a compact form of nickel.

337,019. SYNTHETIC RUBBER. J. Y. Johnson, London. From I.G. Farbenindustrie Akt.-Ges., Frankfort-on-Main, Germany. Application date, May 25, 1929.

Diolefins are polymerised by the action of alkaline earth metals in the presence of organic solvents, and the solution of the polymerisation product is continuously removed. The

(Continued on page 628).

# ROTARY COMPRESSORS AND VACUUM PUMPS *of British Make*

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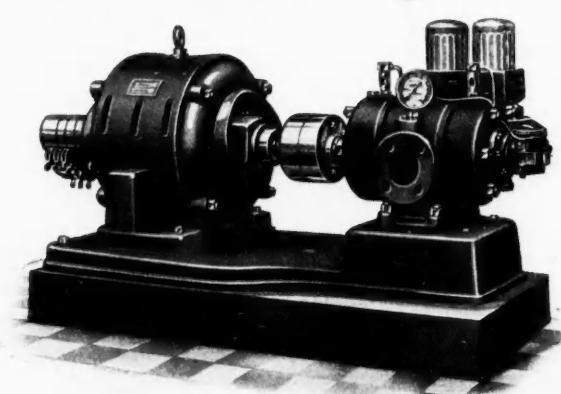
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ROTARY COMPRESSORS & VACUUM PUMPS

diolefine may be introduced into a stream of the solvent before it reaches the catalyst. The solvent may be petroleum ether, ether, cyclohexane, or benzene. The product is suitable for the production of hard rubber.

337,021. DYES. A. Carpmael, London. From I.G. Farbenindustrie Akt.-Ges., Frankfort-on-Main, Germany. Application date, June 21, 1929.

The copper compound of a 1-hydroxy-4-halogen-anthraquinone-2-sulphonic acid is condensed with an alkyl-, aryl-, cycloalkyl-, aralkyl- or acyl-amino compound, the amino residue entering the 4-position. In examples, the copper compound of 1-hydroxy-4-bromo-anthraquinone-2-sulphonic acid is condensed with acetyl-*p*-phenylene-diamine, *m*-phenylene-diamine or acetyl-*m*-toluylene-diamine.

337,028. DESTRUCTIVE HYDROGENATION. Imperial Chemical Industries, Ltd., Millbank, London, R. Holroyd and C. Cockram, Winnington Hall, Northwich, Cheshire. Application date, July 18, 1929.

Bituminous coal is heated *in vacuo* or in an inert gas, or preferably with hydrogen at 250 atmospheres and about 390° C. in the presence of high temperature tar or oils derived from a later stage, and in the presence of catalysts in the form of ribbon, foil, or plates. The coal is deoxygenated and a fusible product obtained, which is further hydrogenated at a higher temperature, e.g., 460° to 470° C. with the same catalyst. The products may be fractionated to obtain petrols and the heavy oils are further hydrogenated or returned to the process. The hydrogen obtained in the second stage may contain 15 per cent. of methane. An example is given.

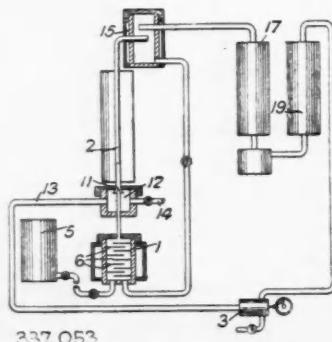
337,046. TREATING HYDROCARBONS. J. Y. Johnson, London. From I.G. Farbenindustrie Akt.-Ges., Frankfort-on-Main, Germany. Application date, July 26, 1929.

Mineral oils, tar oils, oils from destructive hydrogenation operations are converted into oils of lower boiling point employing catalysts consisting of metal compounds of 1:3-diketones soluble in hydrocarbon oils; at a temperature of 300° to 600° C., with or without high pressure. The metal may be tungsten, molybdenum, chromium, vanadium, manganese, uranium, niobium, zirconium, cerium, tin, titanium, aluminium, copper, zinc, cobalt or nickel. Examples of the catalysts include vanadoyl, nickel, chromium or aluminium acetyl-acetonates. Examples are given of the treatment of gas oil and oil obtained from the destructive hydrogenation of brown coal.

337,053. ALIPHATIC ACIDS AND ESTERS. British Celanese, Ltd., 22, Hanover Square, London; and W. Bader, of British Celanese, Spondon, near Derby. Application date, July 27, 1929.

Acetic acid and methyl acetate are obtained by treating a mixture of methanol and inorganic acids containing organic groups or their acid salts with carbon monoxide at 200° to 450° C., and pressure up to 300 atmospheres.

Methanol from a vessel 5 is mixed with phosphoric acid in a chamber 1 at 60° to 90° C., and pass to a gold- or graphite-lined reaction tube 2 heated to 300° to 400° C., into which carbon monoxide and steam are also drawn through pipes 13, 14.



The products pass to a vessel 15 where phosphoric acid separates out, and is returned to the chamber 1, while the vapour passes on to a condenser 17 and absorber 19. Esters in the products may be saponified to produce an acid and an ether.

337,109. PHOSPHORIC ACID AND HYDROGEN. J. Y. Johnson, London. From I.G. Farbenindustrie Akt.-Ges., Frankfort-on-Main, Germany. Application date, August 24, 1929.

Phosphorus or acids of a lower stage of oxidation than phosphoric acid are heated with anhydrous phosphoric acid under pressure. The products are hydrogen and meta- or pyro-phosphoric acid.

#### Specifications Accepted with Date of Application

314,804. Amino-benzyl-ortho-benzoic acids, Preparation of. Newport Co. July 2, 1928.

316,583. Dicalcium fertilisers, Production of. A. Holz. July 31, 1928.

319,273. C-alkyl and C-aralkyl derivatives of aromatic compounds, Manufacture of. Z. Foldi. September 10, 1928.

339,243. Polymerisation products of diolefines, Manufacture of. J. Y. Johnson. (I.G. Farbenindustrie Akt.-Ges.) August 30, 1929.

339,255. Rubberlike masses and articles therefrom, Manufacture of. A. Carpmael. (I.G. Farbenindustrie Akt.-Ges.) August 30, 1929.

339,266. Acid anthraquinone dyestuffs, Manufacture of. A. Carpmael. (I.G. Farbenindustrie Akt.-Ges.) September 3, 1929.

339,267. Vat dyestuffs of the anthraquinone series, Manufacture of. A. Carpmael. (I.G. Farbenindustrie Akt.-Ges.) September 3, 1929.

339,276. Simultaneous reduction of ores and conversion of carbonaceous materials into hydrocarbons of low boiling point. J. L. Fohlen. July 25, 1929.

339,283. Azine derivatives, Manufacture of. A. Carpmael. (I.G. Farbenindustrie Akt.-Ges.) August 30, 1929.

339,291. Cracking of hydrocarbon oils. W. W. Triggs. (Panhandle Refining Co.) June 29, 1929.

339,330. Alkali-metal carbonates and bi-carbonates and soluble magnesium salts, Preparation of. H. E. Potts. (Soc. Anon. Alcalina.) August 1, 1929.

339,332. Compounds of hydrogen peroxide, Manufacture of. J. Y. Johnson. (I.G. Farbenindustrie Akt.-Ges.) August 6, 1929.

339,340. Phosphate rock and the like, Treatment of. Odda Smelteverk Aktieselskapet and E. Johnson. October 27, 1928.

339,348. Cyclic non-saturated ketones with more than nine ring members, Preparation of. Soc. Anon. M. Naeff et Cie. October 9, 1928. Addition to 235,540.

339,352. Vulcanisation of rubber. H. Wade. (Rubber Service Laboratories Co.) September 7, 1929.

339,357. Lead pigments, Manufacture of. Swiss Inventions Syndicate, Ltd., and A. V. Blom. September 7, 1929.

339,359. Basic products derived from higher fatty acids, Manufacture of. I.G. Farbenindustrie Akt.-Ges. August 7, 1929. Addition to 317,325.

339,360. Organic liquids, Treatment of—to facilitate storage and handling. J. Y. Johnson. (I.G. Farbenindustrie Akt.-Ges.) July 4, 1929.

339,371. Cyanates and cyanamides, Manufacture of. J. Y. Johnson. (I.G. Farbenindustrie Akt.-Ges.) September 13, 1929.

339,396. Vat dyestuffs, Manufacture of. A. Carpmael. (I.G. Farbenindustrie Akt.-Ges.) September 28, 1929.

339,410. Mordant dyestuffs, Manufacture of. I.G. Farbenindustrie Akt.-Ges. October 10, 1928.

339,436. Pyridine compounds, Manufacture of. A. Carpmael. (Schering-Kahlbaum Akt.-Ges.) October 31, 1929.

339,469. Aluminum alloy. O. Kamps. November 25, 1929.

339,479. Destructive hydrogenation of carbonaceous materials, Method and apparatus for. W. R. Tate, H. P. Stephenson, J. F. Lehmann, and Imperial Chemical Industries, Ltd. November 29, 1929.

339,489. Halogenated 4:5:8:9-dibenzo-pyrene-3:10-quinones, Manufacture of. I.G. Farbenindustrie Akt.-Ges. December 6, 1928.

339,491. Acetone, Manufacture of. H. D. Elkington. (Naamlooze Vennootschap de Bataafsche Petroleum Maatschappij.) December 6, 1929.

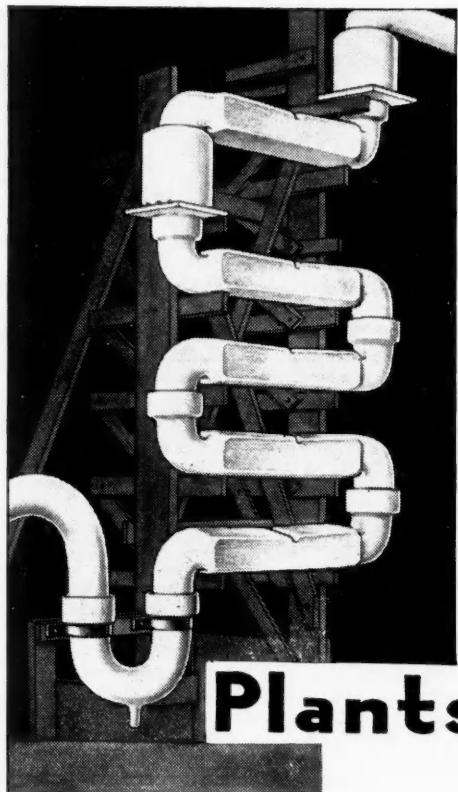
339,504. Magnesium, Preparation of, by electrolysis. C. Arnold. (Dow Chemical Co.) December 16, 1929.

339,516. Dyestuffs of the anthraquinone series, Manufacture of. A. G. Bloxam. (Soc. of Chemical Industry in Basle.) December 24, 1929.

339,521. Heat treatment of manganese steel. H. Wade. (Taylor-Wharton Iron and Steel Co.) December 31, 1929.

339,562. Ammonium nitrate, and calcium carbonate, Production of mixture of. Odda Smelteverk Aktieselskapet and E. Johnson. March 4, 1929.

339,579. Molten mass of metal or alloy, particularly a mass of molten steel in a ladle, Treatment of. F. Krupp Akt.-Ges. April 30, 1929. Addition to 312,063.



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## Commercial Intelligence

*The following are taken from printed reports, but we cannot be responsible for any errors that may occur.*

### Mortgages and Charges

[NOTE.—*The Companies Consolidation Act of 1908 provides that every Mortgage or Charge, as described therein, shall be registered within 21 days after its creation, otherwise it shall be void against the liquidator and any creditor. The Act also provides that every Company shall, in making its Annual Summary, specify the total amount of debts due from the Company in respect of all Mortgages or Charges. The following Mortgages and Charges have been so registered. In each case the total debt, as specified in the last available Annual Summary, is also given—marked with an \*—followed by the date of the Summary, but such total may have been reduced.*]

ALLIANCE ARTIFICIAL SILK, LTD., Oulton Broad. (M., 27/12/30.) Registered November 29, £113,000 debentures (secured by Trust Deed dated November 21, 1930), present issue £60,222; charged on land and buildings at Oulton Broad, also general charge. \*Nil. April 8, 1930.

CHEMICAL ENGINEERING DEVELOPMENT CO., LTD., London, W.C. (M., 27/12/30.) Registered December 6, series of £750 (not ex.), debentures, present issue £698 10s.; general charge. \*Nil. December 31, 1929.

NEW PROCESS SOAP, LTD., Slough. (M., 27/12/30.) Registered December 5, £1,200 debentures, 3rd series; general charge. \*£9,500. December 31, 1929.

NEWCASTLE-UPON-TYNE ZINC OXIDE CO., LTD. (M., 27/12/30.) Registered December 2, £4,000 debentures, to Branch Nominees Ltd., 15 Bishopsgate, E.C.; charged on land at Birtley (Durham), also general charge. \*£8,000. October 15, 1930.

PHOTOMATON PARENT CORPORATION, LTD., London, E.C. (M., 27/12/30.) Registered December 8, £100 and £100 debentures, part of £400,000; general charge. \*Nil. January 23, 1930.

### Satisfactions

ALLIED CEMENT MANUFACTURERS, LTD. (late SHIP CANAL PORTLAND CEMENT MANUFACTURERS, LTD.), London, W.C. (M.S., 27/12/30.) Satisfaction registered December 2, £29,489, part of amount registered September 2, 1926.

BRITISH ALUMINIUM CO., LTD., London, E.C. (M.S., 27/12/30.) Satisfaction registered December 3, £15,884 4s., registered October 14, 1914.

## London Gazette, &c.

### Companies Winding Up Voluntarily

CLAY PRODUCTS, LTD. (C.W.U.V., 27/12/30.) Creditors' claims by January 31. Members' voluntary winding-up. All creditors have been, or will be, paid in full.

GLOBE OILFIELDS, LTD. (C.W.U.V., 27/12/30.) By special resolution December 15. Stanley Hutchinson, of Finsbury Pavement House, Moorgate, London, E.C.2, appointed as liquidator, and authorised to enter into an arrangement with a new company to be formed for the sale to it of the undertaking and assets.

PREMIER OIL AND FINANCE CO., LTD. (C.W.U.V., 27/12/30.) By special resolution December 15. Stanley Hutchinson, of Finsbury Pavement House, Moorgate, London, E.C.2, appointed as liquidator, and to enter into an arrangement with a new company to be formed for the sale to it of the undertaking and assets.

### New Companies Registered

BRENT MANUFACTURING CO., LTD. Registered December 19. Nominal capital, £1,000 in £1 shares. Manufacturers of and dealers in all kinds of paints, varnishes, enamels, washes and distempers, chemicals, insecticides, germicides and agricultural and horticultural implements, etc. Directors: H. C. Hogbin, Highlands, Osterley Park, Middlesex; H. Fear, J.P., Ferndale, Staines.

SALERNI CARBONISATION PROCESS, LTD., Trafalgar House, 11, Waterloo Place, London, S.W.1.—Registered December 18. Nominal capital, £100 in £1 shares. To acquire any patents, patent rights, brevets d'invention,

licences, copyrights and trade marks relating to the carbonisation, distillation or other treatment of carbonaceous materials, and to carry on the business of manufacturers and repairers of and dealers in power transmission mechanisms and change speed mechanisms, motor vehicles, etc.

UNIVERSAL DETERGENTS, LTD., Canning Road, West Ham, London. Registered December 19. Nominal capital, £100 in £1 shares. Soap manufacturers, etc. Directors: J. R. Booer, F. H. Bailey, J. Hill, W. E. Ross.

## Company News

BRITISH DRUG HOUSES, LTD.—The usual quarterly dividend of 1½ per cent. is announced on the preferred shares.

TARMAC, LTD.—A dividend is announced on the 5½ per cent. (free of tax) cumulative preference shares in respect of the half-year ending December 31, 1930.

YORKSHIRE DYEWARE AND CHEMICAL CO.—An interim dividend of 2½ per cent., less income tax, has been declared.

LOW TEMPERATURE CARBONISATION, LTD.—The report for the year to October 31, 1930, states that sales and trading revenue amounted to £76,549, against £115,069 in the previous period of fifteen months. After deducting costs of coal and carbonising and salaries, etc., and including interest and dividends received, there is £30,475, from which is deducted running expenses, research and experimental work costs, leaving £2,683, against £11,578. Interest absorbed a further £12,000, leaving a debit balance of £9,317, which has been transferred to the balance sheet, making a total debit at profit and loss account of £13,811. The report states that the year was on the whole very unsatisfactory from a trading point of view, largely on account of the further fall in price of by-products. Had the prices remained at the level of even 1927-1928, the crude oil from Barugh and Askern would have realised an additional £37,500. The coalite works erected for the South Metropolitan Gas Co. at East Greenwich is now, it is stated, practically complete, the first battery has been heated up, and production will commence early in January.

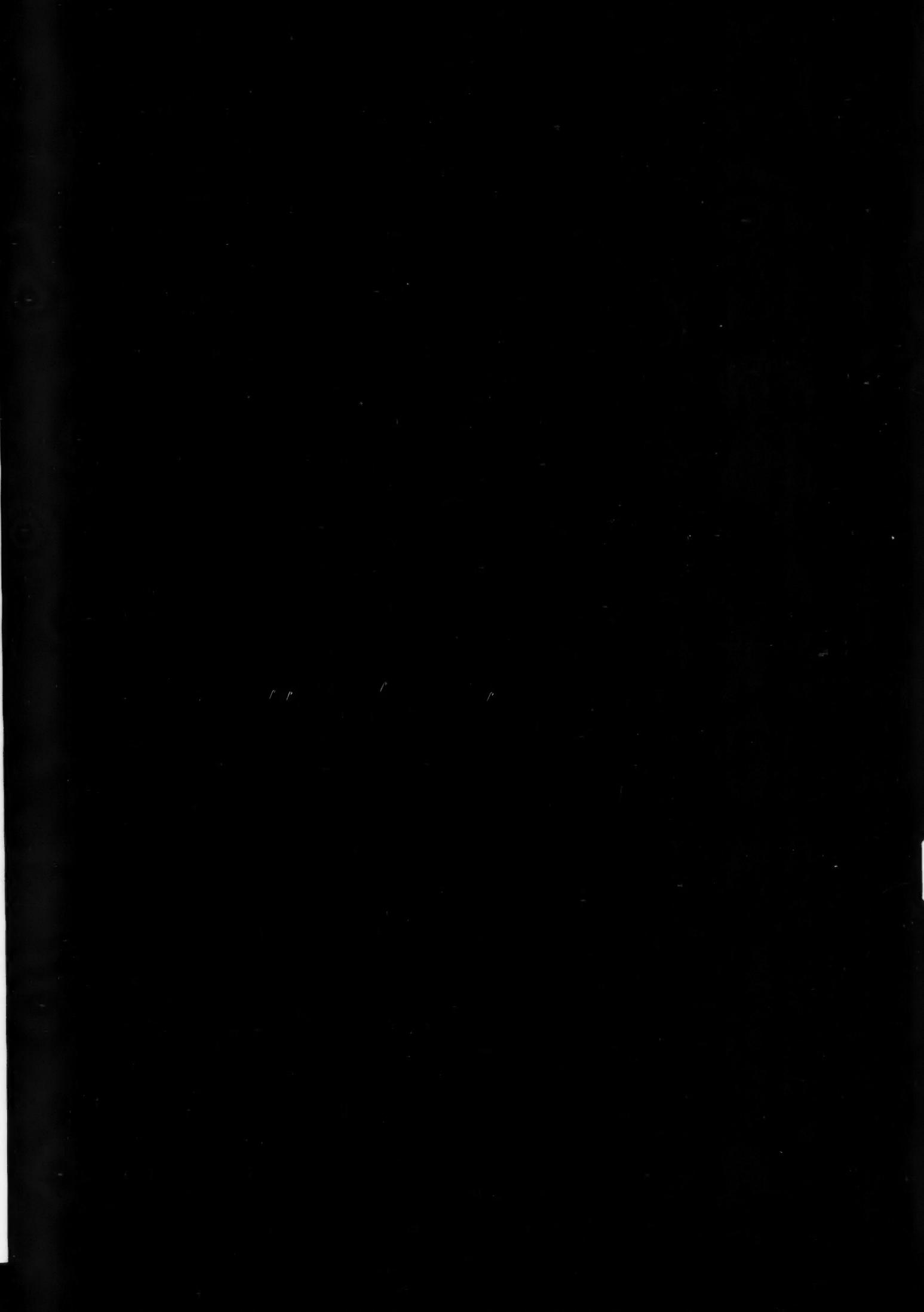
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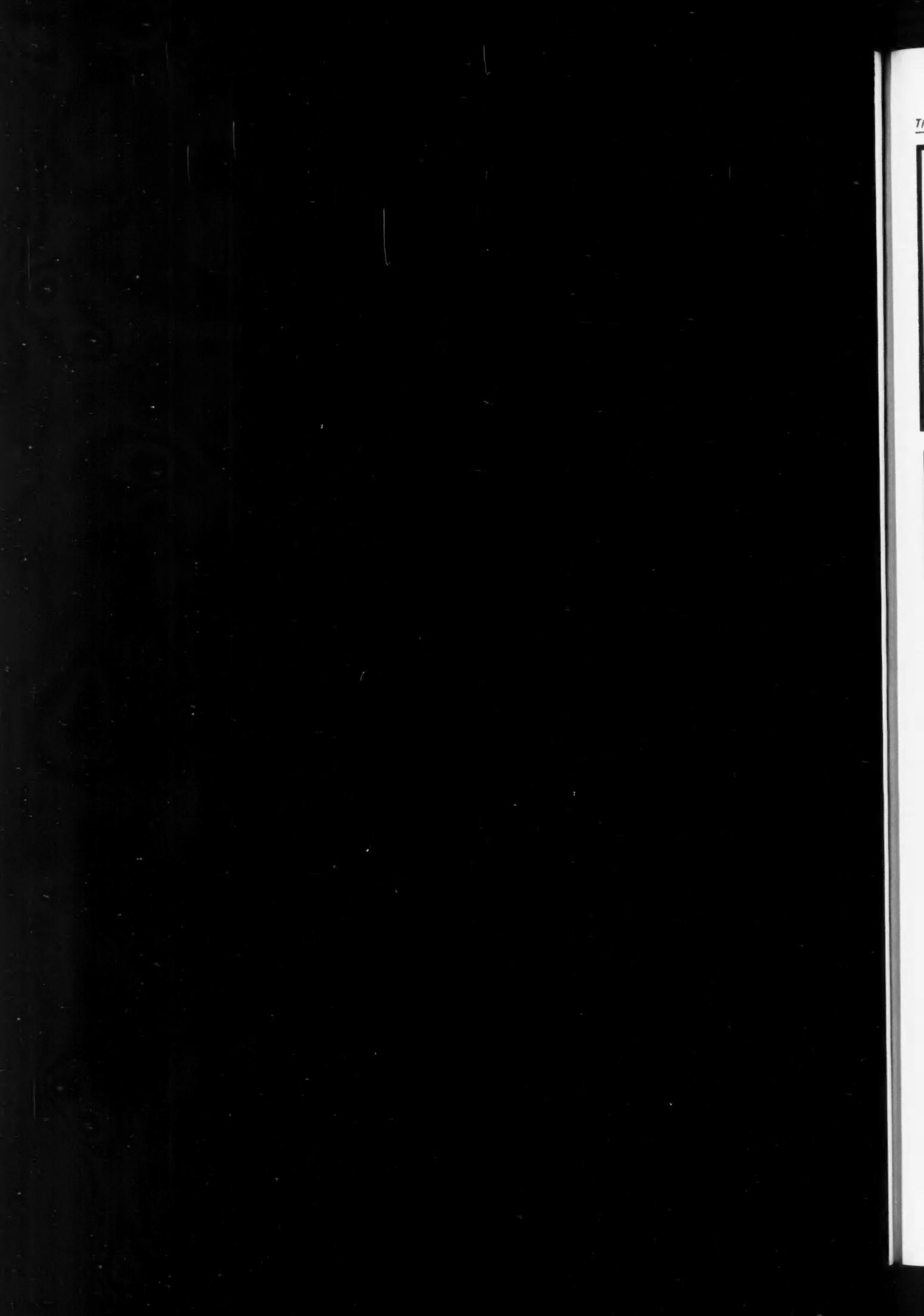
**Refined Coal Tar.**—Like the previous year, the value of this important product fell during April/May at which period it is generally anticipated there will be a firmer tendency. The consumption during the year was lower than normal, but this is accounted for by the change-over in local administration. This change in constitution had the effect of holding up road work very considerably, but it is thought that the coming year will make amends, and therefore a very busy time is ahead for road surveyors and contractors alike. The year commenced with value at about 4d. per gallon, but during April/May, when it was noticed that the demand was below normal, competition became very keen and prices fell to 3½d. per gallon and eased gradually to the present 3d. per gallon.

**Blast Furnace Tar.**—The ironmasters agreed to hold the price of this product at 2½d. per gallon f.o.r. their works in buyers' packages, at which figure it has remained throughout the year. The throughput was steady but rather small.

**Crude Naphtha.**—There was a steady demand for the better grades throughout the year but, nevertheless, quotations fell steadily from about 5½d. per gallon in January/March to about 4d. per gallon at the present time, in sympathy with Solvent Naphthas and Motor Benzols.

**Water White Products.**—Taking the year as a whole the demand for naphthas and benzole has been disappointing. There was no desire on the part of consumers to contract for more than their immediate requirements, with the result that distillers almost invariably had fair quantities of unsold products in stock. Accordingly, values were not forced up and have remained fairly constant. Solvent naphtha 90/160 fluctuated between 1s. 1½d. and 1s. 3½d. per gallon in bulk, ex makers' works, while heavy solvent 90/190 almost always followed suit at about 1d. to 2d. per gallon lower. Motor benzole found a steady, if small, outlet from January to July at round 1s. 5½d. to 1s. 6½d. per gallon, but thereafter the demand fell away and quotations decreased to the present level of 1s. 4d. to 1s. 5d. per gallon.





# The Dyestuffs Monthly Supplement

A Section devoted to the Manufacture and Use of Dyes Published  
in THE CHEMICAL AGE the Second Week of each Month

## LEONIL W

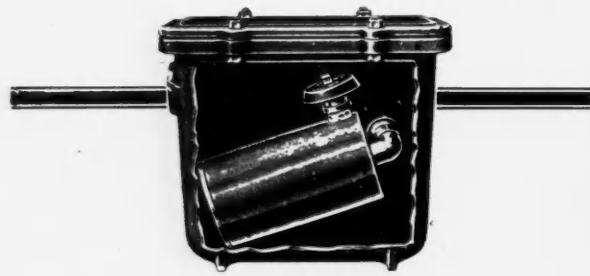
A NEW WOOL SCOURING ASSISTANT OF EXCELLENT  
WETTING OUT AND EMULSIFYING PROPERTIES, WHICH  
CONVERTS THE LIME SOAP INTO A FINELY DISPERSED  
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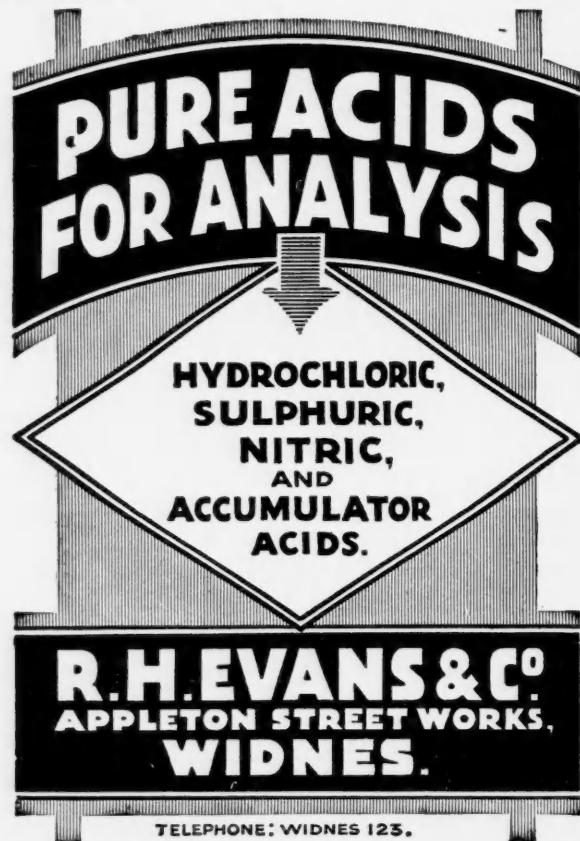
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# Dyestuffs Monthly Supplement

Published in the second issue of "The Chemical Age" each month

Communications relating to editorial matter for the Dyestuffs Monthly Supplement should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Advertisement matter, subscriptions, etc., should be sent to the Manager. The Supplement is devoted to the interests of both manufacturers and users of dyestuffs, and contributions will be welcomed.

### The Castleford Disaster

THE feelings of sympathy evoked by the accounts of the horrors of the explosion at the works of Hickson and Partners at Castleford will be especially present in the minds of colour-users. The dimensions of the disaster are imposing—a death roll of 13 men, scores of houses nearly demolished, a large factory of many buildings laid in ruins. Our condolences go out to the people of Castleford, particularly to the relations and friends of the dead, to the unemployed, to the firm of Hickson and Partners, and to Mr. Ernest Hickson, the founder of the firm, now largely retired from business, ex-President of the Society of Dyers and Colourists, whose work for the Society in many ways and for very many years has been so highly valued by his fellow-members, and in the dyeing industry generally.

### Colour Users' Meeting

The Colour Users' Association are holding their annual general meeting at Blackfriars House, Manchester, at 12 noon on Tuesday next. The annual report and the annual presidential address are usually documents of importance, and this year both may be expected to be at the usual level in this respect. The condition of the textile industry is in itself a matter of grave concern to the users, and the policy to be followed when the Dyestuffs Act runs out at the end of this year is also one of first-rate importance. The time is approaching when some indication of the Government's intentions must be given, and the publication of the report now being prepared on the operation of the Dyestuffs Act in the last ten years cannot be long deferred. In this situation, the attitude of the colour users is an important, if not vital, factor, and the proceedings at Tuesday's meeting will be awaited with interest.

### Natural Dyes of India

Our Indian correspondent sends an interesting article on the revival of the natural dye industry of the country. This, of course, goes back to very early times, but latterly has been all but extinguished by the importation of synthetic dyestuffs. A lady who in 1918 began a special study of the ancient colours of the East, founded a chemical and biological colour and dye research laboratory at Dholpur, and there the production of dyes from natural matter appears to have been studied with some success. The results have been submitted to Bombay and Calcutta colour merchants, and a natural colour factory has been established at Dholpur, capable of a daily output of a ton of colour. So favourable has been the reception of these products that arrangements are being made to market the natural dyes, and it is estimated that in price they can compete with imported synthetic dyes.

### Dyestuffs for Artificial Silk

When experiments began to be made on the production of the new fibres that later became grouped under the general title "Artificial silk" it was little realised what problems would eventually arise when the questions of dyeing and printing came under consideration. It was found to be possible to dye viscose and similar types with the dyestuffs used normally for cotton. At first, it seemed as if there was to be no difficulty. Then, however, weft bars, due sometimes to varying tension in weaving and

sometimes to chemical irregularities, created differences in the affinity of the material to be dyed. For many years the only possible course was to select dyestuffs from the existing ranges as carefully as possible, and until Icyd colours were introduced by Imperial Chemical Industries there was no certain method of covering these inequalities in the dyeing properties. By the production of these Icyd colours at least one of the difficulties was eliminated.

### Cellulose Acetate Silk

Cellulose acetate silk, while possessing many advantages over other types of artificial silk, brought into existence still greater difficulties. It was found that few of the dyestuffs available possessed affinity for this fibre, and the majority even of these produced shades possessing inferior fastness properties. When eventually the Duranol series was discovered, not only were excellent results obtained from a fastness point of view, but, possessing direct affinity for the fibre, these dyestuffs were exceedingly easy to apply. The lead gained in the production of these two groups of artificial silk dyestuffs has not been lost, and continual research and investigation are resulting in the addition of many new dyestuffs to the ranges.

### I.C.I. Booklets

Imperial Chemical Industries are issuing a series of booklets dealing with the dyeing of artificial silks. The first deals with the production of even shades on variable viscose, the second with the obtaining of solid shades on mixtures of viscose and cotton. They are both extremely interesting and cover the subjects carefully from the dyer's point of view. Everybody who uses dyestuffs for artificial silk will find them exceedingly useful. Further booklets are also in hand and will be available at a later date.

The Publicity Department of Imperial Chemical Industries, Imperial Chemical House, London, S.W.1, will be glad to send copies of the booklets to inquirers.

### Dyestuffs Licences for June

The following statement relating to applications for licences under the Dyestuffs (Import Regulation) Act, 1920, made during June has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee:—

The total number of applications received during the month was 514, of which 434 were from merchants or importers. To these should be added eight cases outstanding on May 31, 1930, making a total for the month of 522. These were dealt with as follows: Granted, 497 (of which 471 were dealt with within seven days of receipt); referred to British makers of similar products, 20 (of which 16 were dealt with within seven days of receipt); outstanding on June 30, 5. Of the total of 522 applications received, 487 or 93 per cent. were dealt with within seven days of receipt.

### The Practical Dry Cleaner

*The Practical Dry Cleaner, Scourer and Garment Dyer* (C. Lockwood and Son, pp. 378, 12s.), by W. T. Brant and J. B. Gray, has now reached its sixth edition, and may therefore be taken to have established its usefulness in this large industry. For several years the cleaning and dyeing industry has been experiencing a wonderful growth.

Many new plants have been constructed and older ones remodelled to meet the growing demand. Improvement in equipment and methods has kept pace with the growth of the industry. New processes and machinery are continually being designed, and the chemistry of the subject—a vital section—continues to make advances. The chapter on "Practical Chemistry for the Cleaner and Dyer" illustrates the extent to which a sound knowledge of chemistry enters into industrial processes. The familiar industry of "French dry cleaning" takes its name from the Frenchman, M. Judlin, who in 1866 applied the detergent properties of benzine to the cleaning of fabrics and similar purposes. Even as early as 1848, however, a method of dry cleaning was in use, in which the solvent employed was camphene, an oil of turpentine specially distilled for burning in lamps. The introduction within recent times of new types of materials has added to the difficulties of the practical dyer and cleaner, as it has done in the case of the dyestuff producer, and the cleaning and dyeing business is now a complicated affair compared with what it once was. This text book is certainly not too large for such an industry; it is, although condensed, comprehensive, and its notes on the various sections are clear and practical.

#### **Levies for Wool Research**

Since its incorporation in September, 1918, the Wool Industries Research Association has been supported by voluntary subscriptions. Its activities have so increased that its annual expenditure is now in the neighbourhood of £25,000, and for developments contemplated it is estimated that a minimum sum of £30,000 per annum will be necessary in the future. The Empire Marketing Board subscribes £3,250 per annum, and is also financing an Empire survey of the conditions of wool production. Under the conditions now in operation governing the grant from the Department of Scientific and Industrial Research, the Association is entitled to receive £ for £ up to a maximum of £10,000 per annum in respect of all income raised by the Association in excess of £10,000 per annum. Consequently, in order to earn the maximum grant, the Association has to raise an annual income of £20,000.

A scheme has been devised and approved under which a levy of 2d. per bale will be collected on all imported wool retained for consumption in this country, and a corresponding contribution will be levied on the basis of 4d. per week per operative, payable by the employers in the processing sections, except worsted spinning. Worsted spinners have agreed to share the cost of the levy on raw wool by a payment to topmakers of 3d. per bag on tops delivered. It was estimated that these proposals would result in the following income: Levy on imported wool, £10,482; levy on employed operatives, £10,338; grant from the Department, £10,000; total, £30,000. With the assistance of the shipowners transporting wool from the Colonies to this country, the levy on raw material came into operation on all wool unloaded in this country after 4 p.m. on June 30, and is collected with the freight. In order to make the whole scheme uniform, the levy on the processing sections came into operation as from July 1.

#### **A Review of British Dyestuffs**

Mr. W. E. Moessner, United States Vice-Consul in Manchester, has prepared an exhaustive review of the British dyestuffs industry for the Department of Commerce in Washington, a summary of which is published in the official *Commerce Reports* for June. At present, it is stated, Great Britain produces 10 to 15 per cent. of the world output, and since 1924 exports and consumption have increased 50 and 17 per cent. respectively, while imports have declined more than 30 per cent. Although largely in the hands of Imperial Chemical Industries, there are

still a number of independent firms, among whom mention is made of L. B. Holliday and Co., the British Alizarine Co., Brotherton and Co., British Celanese Co., J. C. Bottomley and Emerson, Williams (Hounslow), J. W. Leitch, British Synthetics, Hickson and Partners, and the Colne Valley Dye and Chemical Co. Of the foreign controlled firms the most important is the Clayton Aniline Co. It is estimated that the total number of persons employed is 11,500. Attention is drawn to the fact that the bulk of the industry's raw materials is now manufactured in this country, and that about 85 per cent. of the home consumption of finished dyestuffs goes into the textile industry.

#### **United States Dyestuffs Production**

Production of domestic dyes in the United States in 1929 was approximately 110,200,000 lbs., an increase of 13,570,000 lbs., or 14 per cent., over production in 1928. The value of the output due to a decrease in prices was less than half that of 1920—the year of maximum value of production. Sales of dyes in 1929 amounted to 104,690,000 lbs., valued at \$44,593,000, or 42·6 cents per lb. Both quantity and value of sales show an increase of 12 per cent. over 1928, according to the U.S. Tariff Commission. In 1929 dyes of domestic production supplied about 92 per cent. of American consumption by quantity and about 80 per cent. by value. There was in addition an exportable surplus of the bulk low-cost colours, principally indigo and sulphur black, of 34,000,000 lbs. The production of vat dyes in 1929 established a new record with a total of more than 9,000,000 lbs., as compared with 6,514,132 lbs. in 1928. The weighted average price of all domestic dyes sold in 1929 showed virtually no change from the average for 1928.

Indigo, the leading colour made in the United States, shows an average sales price of 14·9 cents per lb. in 1929, as compared with 14 cents in the previous year. In 1917, the first year in which domestic indigo was produced, it sold for \$1·42 per lb. The pre-war price, when the entire supply was imported, was 15 cents per lb. The increase in the price of indigo was offset by decreases in the prices of a large number of other dyes. The imports of coal-tar dyes in 1929 were by quantity about 8 per cent. of apparent consumption. The total exports of coal-tar dyes in 1929 were 34,129,597 lbs., valued at \$7,276,618. This represents an increase of 6,305,333 lbs. in quantity and an increase in value of 11·4 per cent. over that of 1928. The exports consist principally of indigo, sulphur black and low-priced bulk dyes. The imports of dyes during 1929 (preliminary figures) were 6,410,355 lbs., with a foreign invoice value of \$5,359,058. This represents an increase of about 20 per cent. by quantity and 24 per cent. by value over that of 1928. Imports originate almost entirely in Germany and Switzerland. The total output of intermediates in 1929 was 349,226,000 lbs., as compared with 279,274,807 lbs. in 1928. Sales totalled 147,418,000 lbs., valued at \$28,145,000, or a unit value of 19·1 cents. In 1928 sales amounted to 115,837,340 lbs., valued at \$24,126,473, or 21 cents per lb.

#### **A New Colour Consciousness**

In the annual report of the U.S.A. Textile Colour Card Association, presented by the director, Mrs. Rorke, it is claimed that the Association has played no small part in the awakening of the United States to a great colour consciousness. "To-day," it states, "our colour, like our music, is an expression of the age we are living in. Through colour hue and form, America is expressing her culture, her sense of the beautiful. It is apparent everywhere—in our architecture, in our homes, in our taste in dress, in our industrial arts. But greater and greater grows the need of trained colourists. Some day, let us hope, we may play an even bigger part in making America a nation of worldwide influence in industrial art as well as in commerce."

## Absorption of Dyes by Cotton and Rayon Materials

By A. J. Hall, B.Sc., F.I.C., F.T.I.

Some practical difficulties in dyeing cotton and rayon materials, arising from the properties of both dyes and textile fibres, are discussed by Mr. A. J. Hall in the following article. He concludes that there exists here a field for much profitable research.

It is a comparatively easy task to classify dyestuffs according to their chemical constitution or affinity for the various fibres and so arrive at generalisations as to methods for their application. But it is necessary to remember that these generalisations are nothing more than generalisations. Practical dyers frequently experience exceptions due to abnormalities in the properties of both dyes and textile fibres. Thus numerous difficulties occur in dyeing, and it is the purpose of this article to discuss some of them.

Most textbooks on dyes or dyeing give reference to theories of dyeing; usually four such theories are cited. But in spite of a large amount of research and effort spread over a number of years, there is at the present time no general agreement as to the process or processes by which textile fibres absorb and persistently retain dyes, and the introduction of new fibres such as viscose and cellulose acetate rayons have only served to make the formulation of a theory more difficult. Curiously enough, it is probably true to say that less interest in such theories is now being taken than formerly. So that the dyers' art is to-day based much less on theoretical considerations than on accumulated and often correlated observations.

### Accessibility of Fibres

In the first place it must be clearly recognised that the absorption of a dye by a textile material is largely determined by the condition and pre-treatment of that material. It makes considerable difference to the dye absorption whether or not the individual fibres in the cotton or rayon yarns composing the textile material being dyed are pressing upon each other tightly. The accessibility of the fibres to the dyes employed is of the greatest importance.

Whenever possible, cotton and rayon fabrics are dyed in the jig, since this type of dyeing machine allows the dyeing process to be carried out rapidly and with the minimum consumption of dye. But it is evident that under such conditions the pressure of the fibres upon each other is considerably influenced. In jig dyeing, the fabric is pulled from one roller to the other in its passage through the dye liquor. The warp threads are thus subject to tension. Since the effect of this tension on a yarn composed of fibres twisted about each other is to cause these fibres to press upon each other more closely, penetration of the dye liquor is proportionally restricted. The tension on the weft yarns is much less. Thus it happens that the warp yarns of a fabric are generally less satisfactorily dyed than the weft yarns.

The dyer must, as far as possible, avoid badly penetrated dyeing, because the dyed materials develop a displeasing character in wear. This defect is not at first noticeable, since the dye is uniformly distributed over the surface of the fibres. But when the material is worn the fibres become displaced relative to each other and the inner fibres, which are white or but slightly stained, become exposed.

### Preparatory Treatment

At least two methods can be adopted for overcoming the difficulty thus caused. The dyer may use the most modern form of jig, in which tension can be reduced to a minimum, or he may specially prepare the fabric being dyed. The first method will not be discussed here except to state that in the modern jig the use of ball bearings, friction clutches, and positively driven guiding rollers, has enabled the tension, and especially irregularities of tension, to be largely avoided. The special preparation involves two factors. Thorough purification of the textile material by previous boiling with alkali and bleaching with active chlorine removes fats and waxes from the cotton and thus makes it more absorbent and less waterproof towards dye solutions. At the same time the alkali-treatment undoubtedly has the effect of permanently swelling the cellulose of the cotton fibres with the effect of increasing its absorbency. It is thus possible to counteract partially difficult penetration of the dye liquor between the fibres by assisting the passage of the liquor into the individual fibres. But the preparatory treatment may act in quite a

different manner. If the fabric is allowed to shrink in the warp during the preparation then the individual fibres in the yarn press with a diminished force upon each other—the yarn becomes more bulky. This effect persists to a certain extent during the subsequent dyeing operation and thus counteracts to a limited extent the prevailing tension.

A striking instance of the value of correct preparation is to be found in the dyeing of fabric consisting of a cotton warp and a viscose weft. This type of fabric is very popular at the present time and is usually dyed in shades of moderate depth by means of direct dyes. If dyed in the usual type of jig it is found, on examination of the dyed fabric, that the rayon is dyed to a much deeper shade than is the cotton. The reasons for this are that the cotton warp yarns are tightly twisted and they are subject to tension during dyeing; further, viscose rayon usually has a greater affinity than cotton for direct dyes. From the point of view of better equalisation of shade on the two fibres it would thus be preferable for the rayon to be in the warp and the cotton in the weft. How can the shades on the fibres be brought more nearly equal? It is found by practical experience that this difficulty can be effectively overcome by scouring and bleaching the fabric in a slack form before dyeing. If the fabric is scoured in a winch machine or by pumping the scouring liquor through it whilst loosely packed in any suitable vat and then dyed in a winch machine, or even if afterwards dyed in a jig, the scouring treatment has so shrunk the cotton yarn as to neutralise the adverse effects of tension in dyeing; the resulting fabric is found to have both types of yarn dyed to the same depth.

### Wetting-out Agents

There are many wetting-out agents on the market for the purpose of assisting penetration in dyeing, and there is no doubt but that they are useful. But a difference must be distinguished between the penetration of the dye liquor into and between textile fibres as present in a yarn and the penetration of the dye particles. It does not necessarily follow that where the dye solvent (water) enters, the dye particles do also. In a yarn composed of cotton fibres twisted tightly about each other these fibres act somewhat after the manner of an edge filter and such a type of filter is very efficient. Diffusion and other experiments have shown that the dye particles in an aqueous solution of a direct dye are usually large, and it would appear that such particles are unable to pass between adjacent fibres. The action of a wetting-out agent probably consists of lowering the surface tension of the solution and also of increasing the dispersion of the dye. The first effect on surface tension is sufficient to ensure penetration of the dye solvent into and between the fibres, but not the penetration of the dye particles. A wetting-out agent only becomes really effective as regards dye penetration if it definitely increases the dispersion of the dye—that is, makes the dye particles smaller such that they more readily pass between the fibres. In support of this it is frequently noticed that a fabric immediately becomes thoroughly wet when placed in a vat dye liquor containing a wetting-out agent, but the penetration of the fabric by dye is subsequently seen to be poor.

The edge filtration effect shown by the fibres in textile yarns is accentuated by the fact that these fibres swell considerably in cross section when wetted. The result of this swelling is a compression of the fibres, thus reducing the space between them and increasing the difficulty of dye particle penetration.

So far, tension on a yarn or fabric has been regarded merely as a means for compressing the individual fibres upon each other. But it has been shown (A. J. Hall, *J. Soc. Dyers and Col.*, 1929, **45**, 78) that the stretching of individual viscose fibres affects their absorption of dyestuffs. Separate viscose fibres, some maintained under tension by means of suitable weights and others allowed to hang slack, were dyed together in solutions of various dyes, and it was found that with certain dyes the stretched fibres dyed to a definitely paler shade than

the slack fibres and with others the difference between the stretched and slack fibres was much less definite. The marked difference of shade was characteristic of the majority of the dyes tested. It is thus evident that in some way or other the stretching affects the fine structure of viscose rayon so as to modify its affinity for dyes. It would be interesting to know if similar effects could be obtained with cotton fibres, but such an investigation is most difficult in view of the small length of individual cotton fibres and also their lack of uniformity.

But the arrangement (as produced by manufacture or preparation) of the individual fibres in the yarns which form cotton and rayon fabrics is only one factor which affects their absorption of dyestuffs. Another important factor is the preparatory treatment which affects the cellulose of such fibres. In particular dry heat, kier boiling, and mercerisation may be cited among such preparatory treatments. These will now be considered.

Generally, the exposure to heat of a cotton or cellulose rayon tends to diminish its affinity for direct dyes. There is also a corresponding decrease in the affinity for moisture—the two affinities run parallel. So that in the singeing of fabrics for the removal of projecting short fibres it is not surprising that the fabric has its affinity for direct dyes decreased. The higher the temperature of heating the greater the diminution of dye affinity. This effect of heating is probably due to two causes—the resulting decreased active surface of the cellulose and the slight chemical changes produced in the cellulose itself. Heating of cellulose in air, as in singeing processes, tends to form oxycellulose, and this has almost no affinity for direct dyes. A singed fabric thus generally dyes to a paler shade than the same fabric not singed.

#### Mercerisation and Kier Boiling

Mercerisation and kier boiling are processes in which the cotton and viscose fibres are subjected to the action of a caustic alkali. The effect is greater in the case of mercerisation because the concentration of alkali employed is very much higher and the temperature of treatment lower than in the case of kier boiling. Both processes have a swelling action on the fibre, and it must be pointed out that in this particular form of treatment a high temperature restricts swelling rather than assists it. Thus viscose rayon can be dissolved in 10 per cent. caustic soda at about  $-8^{\circ}\text{C}$ ., whereas the solution is but partial at  $+15^{\circ}\text{C}$ . The direct result of the swelling is to increase the affinity of the cotton fibre for direct cotton dyes, the connection between swelling and affinity being shown by the similar curves in Fig. 1 (showing increased affinity of cotton for direct dyes as produced by mercerisation) (Pope and Huebner, *J. Soc.*

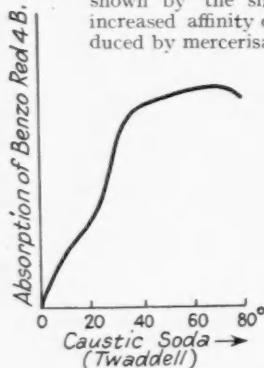


FIG. 1.

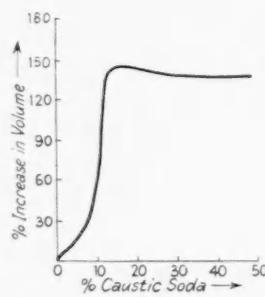


FIG. 2.

*Chem. Ind.*, 1904, **23**, 404) and Fig. 2 (showing swelling of cotton fibres as produced by mercerisation) (Collins and Williams, *J. Text. Inst.*, 1923, **14**, 287).

The effect of swelling as produced by kier boiling and mercerisation is of particular interest as regards materials containing both viscose rayon and cotton. A kier boil of such a material with about 2 per cent. caustic soda does not greatly alter the relative affinities of the two fibres for direct dyes; both before and after the treatment, the rayon dyes to the deeper shade. But after mercerisation with caustic potash (for protection of the viscose rayon), whereby a much

greater swelling is produced, the cotton dyes more nearly to the same depth of shade as the viscose rayon. It thus appears that the cotton is affected to a greater extent than the rayon. This difference is important, for the process of mercerisation thus tends to make the mixed fabric more attractive and assists the dyer. If the dye affinity of the rayon were increased much more than that of the cotton then the mercerisation process for these materials would have to be abandoned.

After mercerisation of cotton goods it frequently happens that they are dried before dyeing. Knecht several years ago found that such drying seriously decreased the affinity of the mercerised cotton for direct dyes, and some indication of the extent of this effect is to be seen in the following results which he obtained (*J. Soc. Dyers and Col.*, 1908, **24**, 67).

| Cotton Yarn.                                 | Absorption<br>of Benzo-<br>purpurine 4B. | Chryso-<br>phenine. |
|--|--|---------------------|
| Per cent.                                    | Per cent.                                | Per cent.           |
| Mercerised and dyed without drying           | 3.24                                     | 0.96                |
| ditto air dried before dyeing                | 3.03                                     | 0.92                |
| ditto dried at $110^{\circ}\text{C}$ . ditto | 2.51                                     | 0.84                |
| Not mercerised                               | 1.77                                     | 0.58                |

The absorption of dyes by mercerised goods is thus largely dependent on their heat treatment between mercerisation and dyeing. It is more economical to dye direct from mercerisation and without drying, but, on the other hand, more even dyeing is obtained if the mercerised material is dried before dyeing. It is sometimes possible to trace uneven dyeing in mercerised goods to uneven drying after mercerisation.

An important application of mercerisation to the dyeing of cotton goods is in the dyeing of Aniline Black. After mercerisation, cotton fabrics have greater absorptive power, such that they are able to absorb a greater amount of an Aniline Black padding liquor. It is thus found that Aniline Black dyed mercerised fabrics have a much better shade than those not mercerised. To-day a large proportion of Aniline Black dyed goods are mercerised solely for the purpose of obtaining the superior black shade thus made possible.

Finally attention will be given to the composition of the dyebath in the dyeing of cotton and viscose rayon materials. Direct dyes are usually the alkali-metal salts of organic compounds containing sulphonate acid groups and are therefore most satisfactorily applied from neutral or slightly alkaline solutions. By addition of small amounts of alkali the rate of dyeing is retarded, but as the alkali—for instance, soda ash—is increased, the absorption of dye by the cotton increases. Thus, one-tenth per cent. of soda ash retards, whilst a dye liquor containing 2 per cent. of the same alkali will dye much more quickly. Similar considerations apply to the effect of the presence of soap.

#### Difference in Behaviour

The absorption of direct dyes by cotton and viscose rayon is largely affected by the state of dispersion of the dye, and this in turn is affected by the temperature of the dye liquor. In this respect the two fibres do not behave alike. Thus at boiling temperatures viscose rayon appears to absorb certain direct dyes to a greater extent than does cotton, but if the temperature is lowered to about  $80^{\circ}\text{C}$ . then the cotton has the greater relative affinity. Such facts as these are of great importance in the dyeing of materials such as knitted goods which contain both cotton and viscose rayon and in which it is desired to dye both fibres to the same depth of shade. It is frequently possible to obtain this degree of levelness by skilful variation of the temperature of the dye liquor during the process of dyeing.

The addition of wetting-out agents to a dye liquor is usually beneficial, but not always in the manner anticipated. The chief advantage to be obtained is level dyeing; this may be anticipated since the material being dyed becomes thoroughly wet as soon as it is immersed. But level dyeing must not be mistaken for well penetrated dyeing, and wetting-out agents do not always secure satisfactory penetration. Sometimes the presence of a wetting-out agent in the dye bath affects the fastness of the dyed materials. For instance, it is claimed that when Nekal BX is employed in the dyeing of indigo the resulting dyeings are faster to chlorine.

The absorption of dyes by cotton and cellulose rayons may be discussed from many angles, but the facts mentioned above indicate that there is much that is as yet not understood. This subject provides a wide field for research.

## Basic Intermediates for Dyestuffs: No. XXXVI.—The Amino Naphthol Sulphonic Acids

By "Consultant"

THE aminonaphthol sulphonic acids fall naturally into two groups, those from 1-naphthol and those from 2-naphthol. These are shown, together with the customary commercial names in the Table below.

From 1-Naphthol—

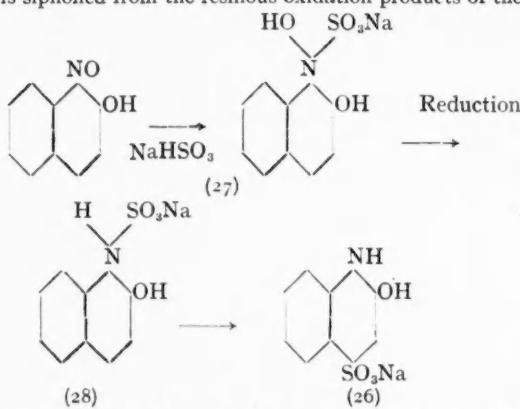
|  |             |
|--|-------------|
| 1-Amino-8-naphthol-4-sulphonic acid      | S-Acid.     |
| 1-Amino-8-naphthol-4:6-disulphonic acid. | K-Acid.     |
| 1-Amino-8-naphthol-3:6-disulphonic acid. | H-Acid.     |
| 1-Amino-8-naphthol-2:4-disulphonic acid. | 2S-Acid.    |
| 6-Amino-1-naphthol-3-sulphonic acid.     | J-Acid.     |
| 7-Amino-1-naphthol-3-sulphonic acid.     | Gamma-Acid. |

From 2-Naphthol.

|                                      |   |
|--------------------------------------|---|
| 1-Amino-2-naphthol-4-sulphonic acid. | — |
|--------------------------------------|---|

Of these, the manufacture of H-Acid was described some years ago in Part I of this series, whilst the acid from 2-naphthol is prepared by a comparatively uncommon process. The 2-naphthol is first converted into the nitroso compound. This may readily be done by dissolving the 2-naphthol (40 lb.) in water (500 gal.) with the acid of caustic soda (½ cwt. 98 per cent. flakes). To the solution so obtained is added sodium nitrite (20 lb.; calc. 100 per cent.) dissolved in a little water at such a rate that the solution does not become warmed; there is no need for artificial cooling at this point. The diazo solution obtained in this fashion is allowed to drop into a vat containing dilute sulphuric acid at  $-5^{\circ}\text{C}$ . at such a rate that the temperature does not rise above  $0^{\circ}\text{C}$ . If the addition is made in the reverse order, the product is said to be lighter in colour. When the final addition of acid has been made, the nitroso-2-naphthol, which has separated as a light brownish powder, is filtered off and washed with water. It is not recommended that more of the product be made at any one time than is immediately required for the next stage in the preparation of the 4-sulphonic acid, since it decomposes on keeping with the formation of deep-coloured substances, due probably to some process of autoxidation.

The conversion of the nitroso compound into 1-amino-2-naphthol 4-sulphonic acid (26) is accomplished by the process of Boniger and involves its reaction with sodium acid sulphite. The sodium sulphite adds on directly to the unstable nitroso group with the formation of a derivative of hydroxylamine sulphonic acid (27). This substance (2-naphthol-1-hydroxylamine sulphonate) is easily reduced by the excess of sulphite present to the amine-sulphonic acid (28) the  $-\text{SO}_3\text{H}$  group of which readily migrates into the "4" position, giving the desired compound. The method of preparation is comparatively simple. The nitroso-2-naphthol (40 lb.) is rubbed up with water in an edge-runner with sufficient water to give a paste. The paste is added to water (20 gall.) and to the mixture, which is replaced in the mill trough, is added 40 per cent. bisulphite liquor (7 gall.) and the milling continued. The nitroso compound goes readily into solution and the clear liquor is siphoned from the resinous oxidation products of the

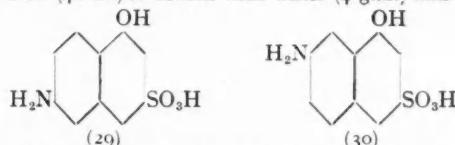


nitroso compound, and warmed in a vat by direct steam to  $40^{\circ}\text{C}$ . 34 per cent. hydrochloric acid being added until the

solution is strongly acid (about 7 gallons). The aminonaphtholsulphonic acid separates on standing and is recovered in the usual way. There is a small but fairly constant demand for this acid, since it gives good developed blacks. The acid is diazotised in the presence of a small quantity of copper chloride and the impregnated fabric may be treated with a m-diamine, when deep blacks are produced on after-chroming.

J- and Gamma-Acids

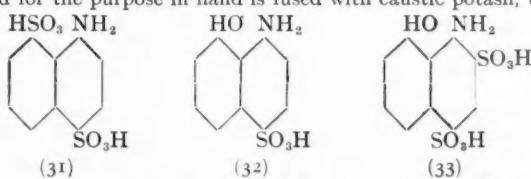
The commercial methods for the production of J- and Gamma-acids, which are useful for direct and developed blacks on cotton, are so similar that they may well be described together. The raw materials are 2-naphthylamine-5:7-disulphonic acid and the corresponding 2:6:8 acid for J- and Gamma-acids respectively. The sodium salt of the disulphonic acid (40 lb.) is heated with water (4 gall.) and caustic



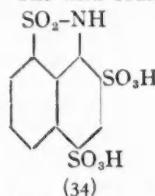
soda (40 lb.) in a steel autoclave fitted with a stirrer at  $200^{\circ}\text{C}$ . for six hours. After cooling, the contents of the autoclave are chipped out, dissolved in water and the solution made strongly acid with mineral acid when the required acid is precipitated. The J-acid (6-amino-1-naphthol-3-sulphonic acid) (29) is only sparingly soluble in water, as also is the Gamma-acid (7-amino-1-naphthol-3-sulphonic acid) (30). Neither acid presents much difficulty in its preparation.

S-Acid and 2S-Acid

The preparation of the 1-naphthylamine-4:8-disulphonic acid (31) which is the raw material for the production of S-Acid, has been described in an earlier number of this series, and for the purpose in hand is fused with caustic potash, the



proportion of the latter being five times that of the acid used. The replacement of caustic potash by caustic soda in this process decreases the yield, although no reason has been advanced for this decrease. In addition to the potash, water is used (one gallon to each ten pounds of acid) and the melt is continued until a sample, taken out and diluted with water, shows the green fluorescence which is characteristic of the sodium and potassium salts of the acids of this group. On diluting the melt with its own weight of water and acidifying with hydrochloric acid, the S-Acid separates. The same 4:8-naphthylamine disulphonic acid is used as the starting point for the production of 2S-acid, save that in this case it is converted into the sultam of 1-naphthylamine-2:4:8-trisulphonic acid (34). The acid sodium salt of the 4:8-di-

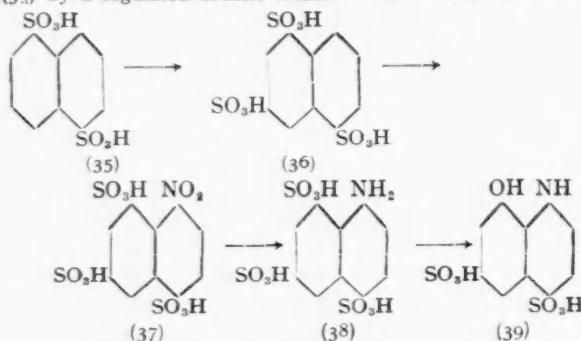


sulphonic acid is added slowly and with good stirring to twice its weight of 40 per cent. oleum, and the solution gradually warmed to  $80^{\circ}\text{C}$ . at which point the formation of the sultam starts and solid separates until the stirrer is almost stopped; eventually, a solid mass is obtained but the heating is only

discontinued when a sample diluted with sodium acetate solution gives no diazo reaction, nor couples with diazo-p-nitroaniline. When this point has been reached it is certain that the maximum yield of sultam has been produced, and the contents of the pan are diluted with water and the acid isolated by the process of conversion to the calcium and sodium salts successively, the final salt being evaporated to dryness. The process for the conversion of the sultam-disulphonic acid to the 2S-acid is very similar to that already described in the preparation of 2S-acid. The caustic fusion succeeds better with potash than with soda, as before, and the proportions of acid, potash and water are slightly different from those mentioned in that preparation. For the preparation of 2S-Acid four parts of the sodium salt of the naphthasultam acid are mixed with ten parts of the caustic alkali and one part of water, and the fusion is conducted at 170° C., at which temperature the sultam rapidly dissolves, and when there is no further reaction (corresponding to no increase in colour of a diazotised and coupled sample) the melt is poured out into water and the 2S-acid (33) precipitated by the addition of hydrochloric acid.

#### K-Acid

The raw material for the preparation of K-Acid is naphthalene-1 : 5-disulphonic acid (35), which is sulphonated to the 1 : 3 : 5-trisulphonic acid (36), nitrated to the 1-nitro-4 : 6 : 8-trisulphonic acid (37), reduced and converted to the K-acid (39) by a regulated caustic fusion. There are several dif-



culties in the way of the process; there is danger during the sulphonation of the 1 : 5-disulphonic acid of the latter being transformed into the isomeric 1 : 6 and 2 : 7 acids, so that a low temperature is necessary to avoid this. Further, to reduce the possibility of this isomeric change to a minimum, 70 per cent. oleum is used. In actual practice the process follows so nearly that which was given in Part I of this series for the preparation of H-Acid, that a further repetition of the details is unnecessary.

#### British Foreign Trade in Pigments

BRITISH official returns of the import and export trade in pigments for 1929 indicate quantity increases for all classes except exports of dry white lead. Comparative figures for the last three years were:—

|                    | IMPORTS   |           | 1928      |           | 1929      |           |
|--------------------|-----------|-----------|-----------|-----------|-----------|-----------|
|                    | Cwt.      | Value     | Cwt.      | Value     | Cwt.      | Value     |
| Barytes, etc. .... | 822,666   | \$804,286 | 825,774   | \$799,323 | 831,003   | \$782,290 |
| White lead, dry .. | 166,850   | 1,109,864 | 189,884   | 1,248,077 | 185,920   | 1,301,576 |
| All other sorts .. | 1,252,358 | 7,382,135 | 1,414,642 | 8,148,146 | 1,433,478 | 8,800,447 |
|                    | EXPORTS   |           |           |           |           |           |
| Barytes, etc. .... | 17,939    | 36,718    | 42,538    | 82,653    | 54,964    | 119,799   |
| White lead, dry .. | 61,438    | 495,317   | 58,837    | 478,031   | 54,139    | 464,580   |
| All other sorts    | 731,839   | 6,036,611 | 711,179   | 5,683,571 | 719,140   | 5,630,001 |

#### Swiss Dye Trade

SWISS exports of aniline dyes in May totalled 581,866 kilogs valued at 5,636,232 francs, against 557,206 kilogs valued at 5,273,668 francs in April, and 646,982 kilogs valued at 6,324,459 francs in May, 1929. German takings amounted to 128,794 kilogs, which compares with 130,379 kilogs in April. The second most important market was Great Britain, sendings to which country improved from 44,374 kilogs in April to 71,788 kilogs in May. Czechoslovakia with 47,367 kilogs against 47,770 kilogs, France with 41,216 kilogs against 45,474 kilogs, and Italy with 29,002 kilogs against 31,372 kilogs, all registered declines. Japan, Belgium and Spain all increased their business.

#### The Indian Natural Dye Industry

[FROM OUR INDIAN CORRESPONDENT.]

THE colour and dye industry of India is a thing of olden times. With the solitary exception of indigo-dyeing, the old processes are practically extinct, yet at one time India was foremost in the art of dyeing. Slight signs of the industry may still be seen in some of the jails where Indian vegetable colours are used in the manufacture of carpets and durries. The old Maheers, who alone could have kept alive the ancient traditions, are dead and gone. Their descendants are eking out a miserable existence in different parts of India, and partly owing to their poverty and partly to the introduction of artificial dyes, have forsaken the work for which their ancestors were renowned.

These vegetable dyes have now been replaced by the use of imported aniline dyes, which are very popular on account of the low price and ease of use, thus saving those concerned the trouble of extracting colours from the Indian flora. The average Indian dyer of to-day does not appear to care whether the imported dyes are as good as the ancient vegetable dyes, so long as they enable him to earn his livelihood. Hence, all interest in the old dyes is gone, although this art was handed down from father to son from time immemorial, and so the ancient colours for which India was famous are no longer made.

#### India's Resources

India's indigenous dyes have been entirely supplanted by artificial dyes of foreign origin. Seeing the great profits of this industry, America, Japan, Switzerland as well as Great Britain commenced the manufacture of aniline dyes. All of these countries are exporting their dyes to India. The aniline or coal tar colours were known to India in the past. This is proved by the recipes obtained for manufacturing the colours, though in a crude form, not as a by-product but from coal itself.

Great are the resources of India with respect to the dye industry, since the old generation of dyers was able to manufacture the colours from almost any plant growing in their own districts. Hence, the processes that were known to them are varied and the colours differed in shade in various parts of India as one plant was substituted for another. The lack of any particular plant was no drawback, as many others could be used, these giving a dye which only varied slightly in shade, the quality of the dye being quite as good.

Mrs. Hayat E. Hate, a Georgian lady, having adopted India as her home, was strongly attracted by the brilliancy of the ancient colours of the East, and in 1918 began a special study of them with her husband, Colonel Hate. At great personal expense, research work was carried on, and it is claimed that almost all the lost knowledge of this art was regained.

#### Research Laboratory at Dholpur

The next step was the founding of India's Chemical and Biological Colour and Dye Research Laboratory at Dholpur, where experiments were carried out by Mrs. Hate with her husband's assistance, which proved that the matter they had collected from all parts of India was really satisfactory and useful for promoting the dead colour and dye art of ancient India, and thus reviving the time-honoured industry of the country, and giving a fresh impetus to the advancement of commerce and industry in India in this direction.

Products of these experiments were taken to Bombay and demonstrations given to some of the leading colour, chemical dyeing and bleaching merchants of the city, who are more or less satisfied with these genuine vegetable dyestuffs. Having convinced them of the great possibilities of manufacturing these colours for the benefit of the country, Mrs. Hate, with the help of friends, has started a colour and dye manufactory at Dholpur which is capable at the present time, in its infancy, of a daily output of one ton of colour.

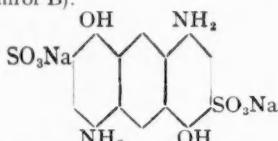
Satisfactory demonstrations have been given in Calcutta by the factory's chief representative to some of those interested in the colour and dye industry, and arrangements are being made to market these dyes without delay. With regard to the cost of manufacture, this will allow of the sale of indigenous dyes at a price successfully competing with the price of imported dyes.

## Dyes and Their Application: Recent Technical Progress

By L. J. Hooley

## Acid Alizarine Colours

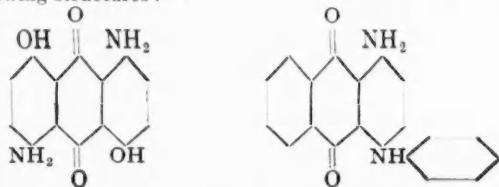
WHILE the hydroxy anthraquinones, such as alizarines, are insoluble and employed as mordants, they can, of course, be made soluble by sulphonation as in Alizarine Red S; such sulphonated derivatives are not, however, in the case of the lower hydroxy derivatives at any rate, nearly so important as the unsulphonated ones, nor, as a matter of fact, does the sulphonation do away with the necessity for a mordant, since normally the mordant is necessary either to give the desired shade or fastness. With some of the more highly substituted hydroxy anthraquinones, however, particularly those which also contain amino groups, the sulphonic acids become important in themselves and are used as such instead of with mordants. These sulphonated acids are remarkable as giving great brightness combined with excellent fastness to light. This was first discovered in the case of the body shown below (Alizarine Saphirol B).



This body met with immediate success when put on the market. This success, as usual, led to a search for homologous members, and as a result a range of red to black was brought out, the range including Alizarine Irisol, Rubinol, Astrol, Cyanine Greens, Cyananthrols, etc., acid colours of remarkable brilliance and fastness. The blues have, of course, been the most important and form the fastest of the various acid direct blues. The greens come next in importance

## Blues

The acid alizarine blues fall roughly into two classes: (a) the diamino dihydroxy anthraquinones, such as the Saphirols, and (b) the amino arylido anthraquinones as Alizarine Sky Blue. The colour bases have essentially the following structures:

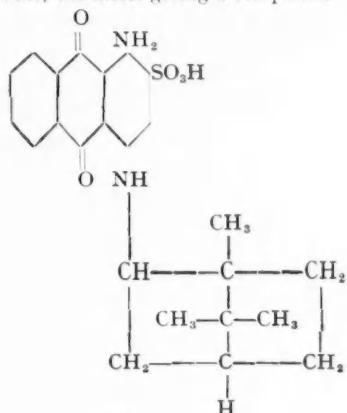


and in so far as the difference in structure gives differences in properties, (a) tends towards increased solubility, good levelling power and elasticity in dyeing, while (b) gives better fastness to chrome and salt. The ease of dyeing is seen at its best in the disulphonic acid of (a) already mentioned, and manufactured as Solway Blue B. Many attempts have been made to synthesise new bodies combining the merits of both groupings, but only with limited success. The majority of the new bodies belong to type (b). Alizarine Sky Blue B was the 1-amino-2-bromo-4-p-toluidido-2-sulpho derivative. Instead of having the sulpho group in the p-toluidido grouping it may replace the bromine in the 2-position, and various dyes having constitutions such as may be obtained in this way were first synthesised about 1914, and continue to appear in new patents. Some of the more interesting of these will be mentioned below:

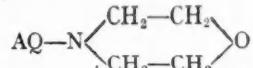
In B.P. 280,637 (I.G.F.A.-G.) 1-amino-4-halogenanthraquinone-2-sulphonic acids are treated with alkyl or aralkylamines in the presence of catalysts. In this way, for example, a body of Sky Blue type is obtained which has an alkyl or aralkyl group in place of the usual aryl. Only where the molecular weight of the amine is high enough will a blue be obtained, methylamine, for example, would give a violet. The patent gives examples with normal butylamine and benzylamine, condensation in the case of the former being done in aqueous solution at 70-80° C. with copper sulphate as catalyst.

According to B.P. 276,408 (I.G.F.A.-G.), the clearer shades obtained by the use of aliphatic amines such as those mentioned in the patent just discussed are offset by the fact that

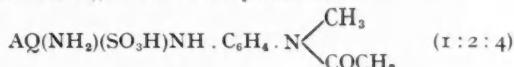
the fastness to light is not so good as with arylamines. This disadvantage is claimed to be overcome by using hydroaromatic amines, i.e., those which double the functions of aliphatic and aromatic. The patent is a general one, claiming the introduction of hydroaromatic amine residues into anthraquinone derivatives by customary methods. Among the examples, is the treatment of 1-amino-4-bromo-anthraquinone-2-sulphonic acid with hexa hydroaniline, using sodium carbonate and copper sulphate, to give a clear blue dye. Closely related to and coming within the above two patents is the process of B.P. 282,452 (I.G.F.A.-G.), which describes the preparation of amino anthraquinones in which one atom of the amino group is substituted by a cyclic paraffin such as pentamethyleneamine or bornylamine, the latter giving a compound



Again somewhat similar is B.P. 317,355 (I.C.I., A. J. Hailwood, W. W. Tatum and G. E. Watts), where the amine used is morpholine, although, like the preceding patents, the process is not necessarily primarily directed to blue acid dyes. With 1-chloranthraquinone a red body having affinity for acetyl silk is obtained:



Instead of replacing the arylamine group by an alkylamine or similar one, substituted arylamines may be used, especially substituted aminoarylaminies. In a recent patent, B.P. 302,928 (I.G.F.A.-G.), m-aminomethyl acetanilide is used:



Instead of m-aminomethyl acetanilide, other acylated m-phenylene diamines may be substituted. The corresponding p-derivatives are mentioned in earlier patents. The use of such amines is claimed to give greater solubility as compared with unsubstituted ones.

## Green Acid Dyes

To obtain a shade as deep as green, it is usually necessary to have two arylamine groups para to each other in the anthraquinone molecule, as in the simplest member 1,4-di-p-toluidido-anthraquinone sulphonic acid (Solway Green). Aliphatic or hydroaromatic groups are not sufficient to give green shades. The reason for this is not clear; if the red violet 1,4-diamino anthraquinone be compared with the blue 1,4-dimethylamino anthraquinone it might be expected that the tetramethyl body would be blue-green or green, but it is only blue (although much more basic). Similarly the di-propylamino anthraquinone is only blue; this is not because the propyl group has too low a molecular weight, as can be seen by taking the di-hexahydroanilido derivative, which is still only blue, while the lighter dianilido is blue-green.

The 1,4-diarylamine anthraquinones form exceptionally fast green acid dyes after sulphonation, their fastness to the usual agents being generally excellent. They are not, how-

ever, as important as the acid alizarine blues, as the demand for green shades is so much less, and because greens can also be made from the blues by shading with a fast yellow. These greens are usually synthesised by the action of an amine on a leuco quinizarine or on a 1,4-dichloranthraquinone derivative, and while condensation takes place readily enough with the 1,4 derivatives themselves there is sometimes difficulty with substituted 1,4-derivatives. For example, in synthesising 1,4-dianilido-5,8-dihydroxy anthraquinone from the leuco 1,4,5,8-tetra-hydroxy, there is trouble through resinification, but this may be avoided, according to B.P. 263,370 (I.G.F.A.-G.) by adding arsenic acid to the condensation melt. In the alternative way of preparing the same body from aniline and 5,8-dichlorquinizarine by replacing the chlorine, a copper catalyst unexpectedly leads to an unsatisfactory result, but by its omission good greens can be obtained (B.P. 244,450, I.G.F.A.-G.). Rather elaborate conditions for condensing 1,4-dichloranthraquinone-5-sulphonic acid with p-toluidine-0-sulphonic acid are detailed in U.S.P. 1,580,265 (Marvine), silver or mercury being recommended as a catalyst.

## A New Polish Laboratory Washing Machine For Determining Fastness of Dyeings

The "American Dyestuffs Reporter" publishes the following description of a new Polish laboratory washing machine, known as the "Pralka," for determining fastness of dyeings to washing, water, acids, alkalis and perspiration, for which a patent has been applied for. It is stated to have received the approval of the Polish Fastness Commission.

The Pralka apparatus has proved to be extremely convenient, and has given accurate results. On experimental trial the apparatus shows, on the washed and untouched places, differences easily observed.

At the meeting of a sub-committee to test the fastness of dyeings, the Pralka was very flatteringly received. The apparatus is entirely constructed in Poland.

### Description

The essential part of the apparatus consists of a double ring in two parts, accurately fitted to each other, 8 cm. on the outside and 5 cm. inside diameter. This ring has a perpendicular movement with a turn of 10 cm. and a rate of 60 turns in a minute. The ring with the sample moves in an experimental bath, and, at the highest position of the turn, rises a little above the surface of the liquid. In order to keep the temperature of the experimental bath to the proper level, the vessel is put in a steam bath which is heated either with a Bunsen burner or by an electric heater or any other convenient means. The vessel is covered with an easily-opened cover, which prevents the escape of too much foam and its squirting on all sides. The whole apparatus is solidly nickelled, and the vessel for the experimental bath as well as the rings is made of nickel plate, due to which the experiments can be made under the influence of weak acids. The apparatus is of a very light, but solid, construction. Its weight is 60 kg., height 110 cm., and base 60 by 60 cm.

### Method of Testing

In order to perform the test, one cuts by means of the model which is added to the apparatus, round pieces of the trial fabric, some pieces of white cotton web, and a woollen one, fixing them closely with thumb nuts into the ring, arranging the trial coloured sample between the white samples. Loose fibre and yarn are tested in the same manner, and such samples are pressed between the two round pieces of the white fabric. The testing liquor is poured direct into the vessel. A normal bath contains one litre of fluid, the amount being indicated by the key on the horizontal tube. When it has been brought to the proper temperature, by means of an easily disjointed coupling the ring with the sample is fastened to the operating parts of the apparatus, and then the protecting cover is pushed in front, the motor is attached, and the apparatus works automatically. It is necessary only to control the temperature of the bath during the testing. After the experiment is completed, one opens the cover, and the ring is easily taken off, the samples are taken out, thoroughly rinsed, dried at a low temperature and preserved as a record. The pouring out of the bath is done through a special cock. The fluid is poured into the hot water bath through the tube of the levelling device.

### Dyestuffs Trade in Scotland

REPORTS for June indicate increased activity as compared with previous months. Bearing in mind recent tendencies, this change is more reassuring and suggests that perhaps an unduly pessimistic view of the trade outlook can be taken. Stock Exchange prices are undoubtedly responsible for much of the present feeling, but the falls in prices in many cases have undoubtedly been sympathetic. Numbers of industrial stocks have fallen below their real values, and as there has been nothing in trade conditions to justify the drop, improvement in feeling may show a recovery to prices which are nearer the proper ones. Recent events have indicated how quickly opinions may change on the protection question, and future months appear to hold almost anything in store.

The woollen trade has shown little alterations as compared with last month, and dyers and finishers are still suffering from inadequate orders. Dyestuffs sales have shown small rises, and throughout the trade generally on balance there appears to be an improvement.

The accurate working of the washer and the fully regulated mechanical parts of the process permits one to state quite accurately the fastness of the dye. Accuracy of the washer requires that distilled water be used in making up the testing solution, because differences in the water, whether it is hard or soft, may have an influence on the results obtained. An exact and detailed classification of the fastness of dyes within a reasonable number of groups may be obtained. The washer is so accurate that in dyeings, for example, with benzopurpurine, one is able to find on the pressed parts of the sample the degrees of running of the colour. It is likewise possible to register the changes of shades under the influence of the reagent.

The result of the examination is not obtained by virtue of the dyeing itself becoming weaker, but by the dyeing of the white fabric, which is held in control with the sample being washed. It proved convenient to indicate the grade of fastness according to the first signs of coloration of the white fabric. Owing to the arrangement of washing the sample by immersion and removal, one succeeds in catching the first signs of colouring at the top of the stroke. A further control consists of the colour of the soap bath.

With such indications, erroneous comparison with other dyeings is removed. For example, we give the average fastness of dyeings on cotton to washing, such as has been proposed to the sub-committee for standardisation of colour fastness of the Polish Fastness Commission.

Reagents: Distilled water, neutral Marseilles soap, soda and soda oil, 38° Bé. :—

Class 1.—Distilled water, 15° C., 15 minutes.

Class 2.—Distilled water, 15° C., 60 minutes.

Class 3.—Distilled water plus 2.5 per cent. soap, 15° C., 60 minutes.

Class 4.—Distilled water plus 2.5 per cent. soap plus 2 per cent. soda, 15° C., 60 minutes.

Class 5.—Distilled water plus 2.5 per cent. soap, 30° C., 15 minutes.

Class 6.—Distilled water plus 2.5 per cent. soap, 45° C., 15 minutes.

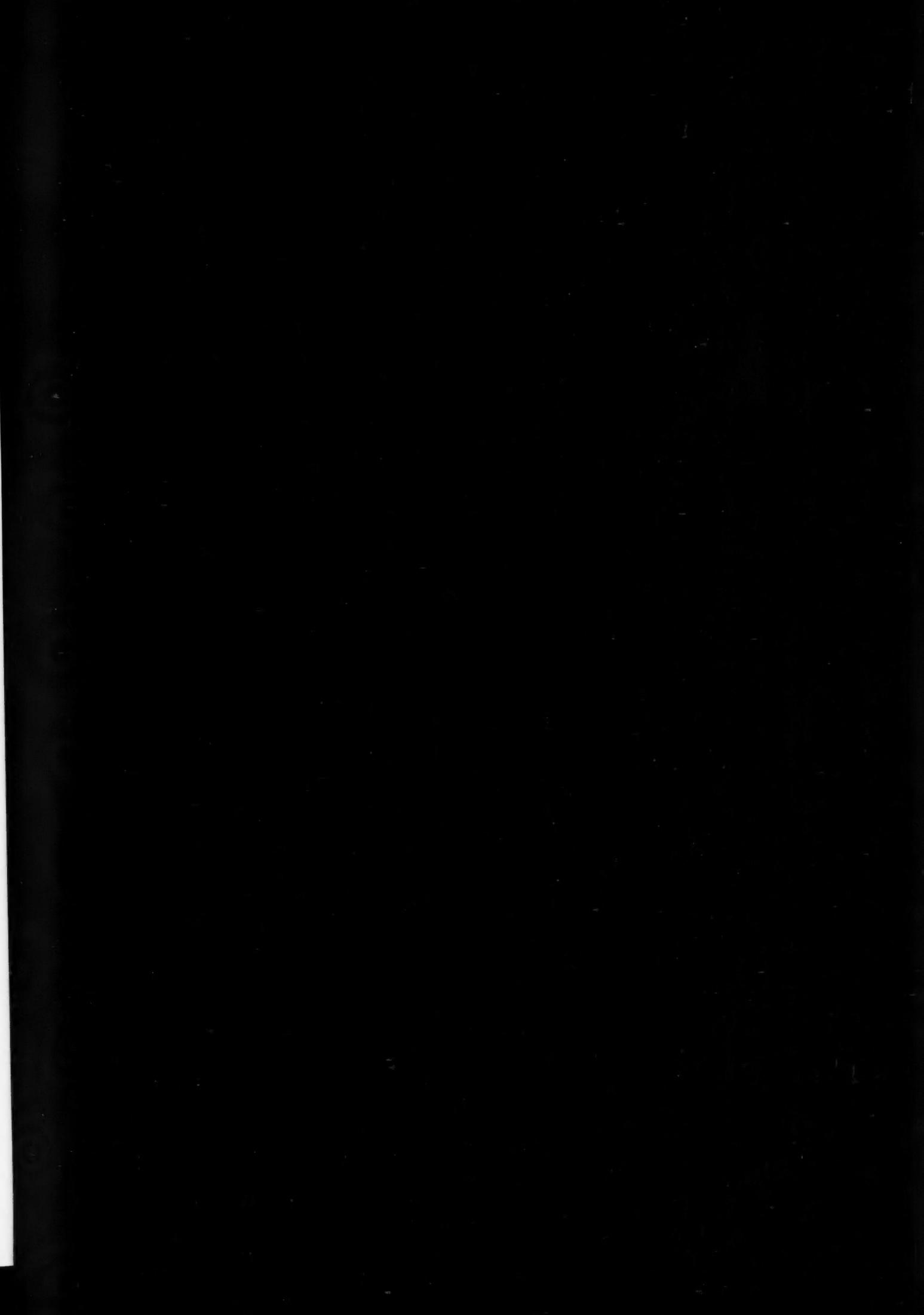
Class 7.—Distilled water plus 2.5 per cent. soap plus 3 per cent. soda, 60° C., 30 minutes.

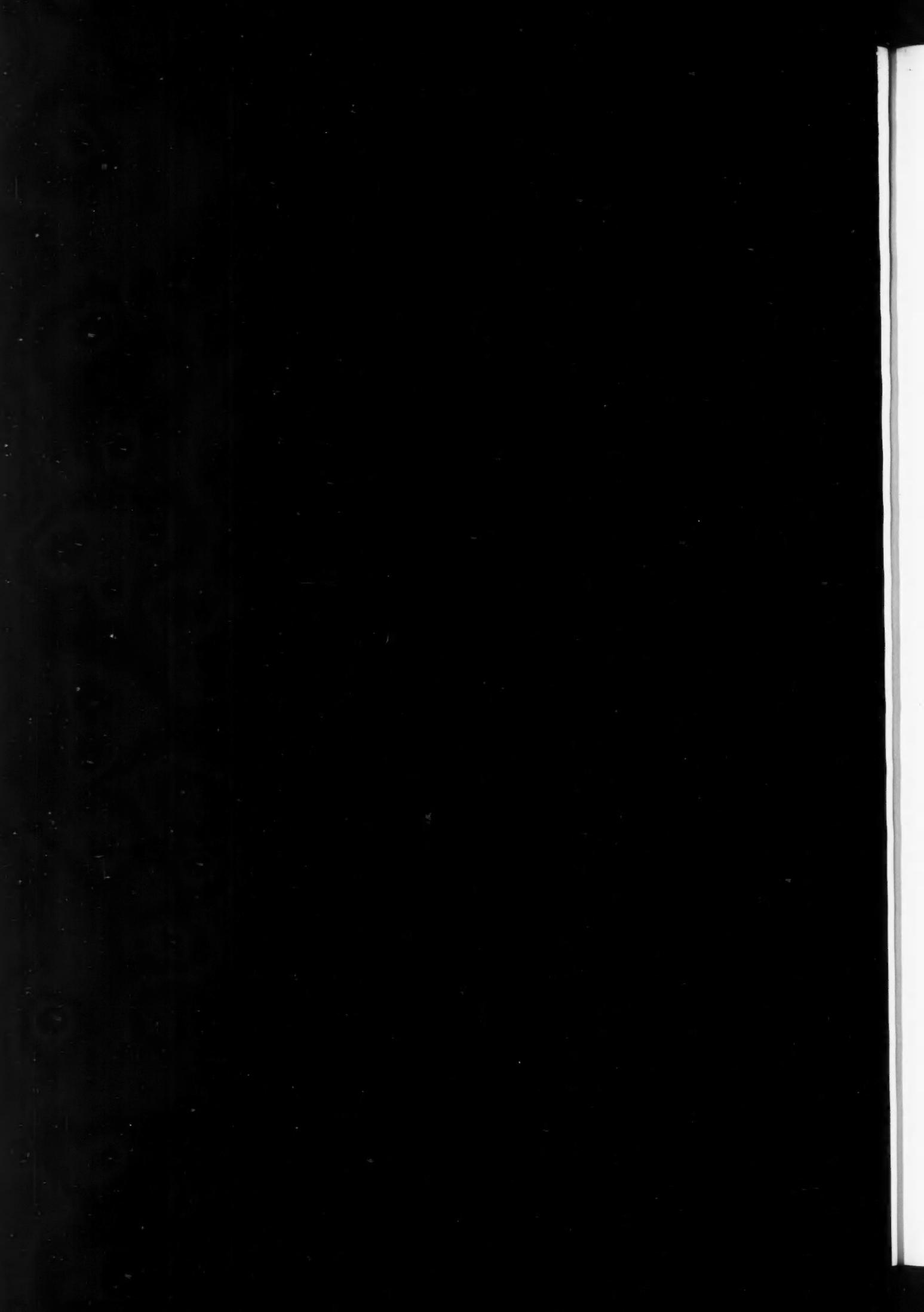
Class 8.—Distilled water plus 2.5 per cent. soap plus 1 per cent. soda oil, 95 to 100° C., 30 minutes.

The fastness of the dye is placed in the highest class, in which the white samples washed together with the dyeing remain quite uncoloured or unstained.

With similar precision in the same apparatus, examinations as to the fastness of dyes in water, perspiration, acids and alkalis can be performed.

The apparatus is made by the Technika Gorzelnica Co., Warsaw, Poland.





# Dyestuffs Monthly Supplement

Published in the second issue of "The Chemical Age" each month

Communications relating to editorial matter for the Dyestuffs Monthly Supplement should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Advertisement matter, subscriptions, etc., should be sent to the Manager. The Supplement is devoted to the interests of both manufacturers and users of dyestuffs, and contributions will be welcomed.

## The Dyestuffs Act

The industry is still without any definite statement of the Government's intentions as to the Dyestuffs (Import Regulation) Act which, in the normal course, will expire early in January next. From an answer in the House of Commons reported in our last issue, it was apparently intended to make an official announcement before the House rose, but the President of the Board of Trade on Thursday week stated that it would be impossible for any announcement of Government policy to be made before Parliament reassembles. It may be inferred from the terms of Mr. Graham's answer that the Government already have before them the report of the Dyestuffs Development Committee on the working of the Act during the past ten years, and that consultations are proceeding between the Government and the Committee. It is reasonable to expect that the recommendations of the Committee will have weight with the Government, but what they are is not yet known.

Mr. Graham's latest answer to questions makes it clear that the Government have not even yet reached any decision. One can imagine that opinion within the Government may be divided, for the future of the Dyestuffs Act will be a definite test of their general attitude, but whatever the final policy may be that they decide on, it is desirable that there should be as little delay as possible in making it known. The present uncertainty leaves British manufacturers in a state of anxiety. Even if, as some are inclined to think, the degree of protection afforded by the Act may be modified, it would be an advantage to know some time in advance so as to be able to prepare for new conditions. In addition to the dyestuff manufacturers, the users are naturally interested in this question, and the merchanting class at home as well as competitive makers abroad are watching the situation expectantly. At present there is much speculation as to what may happen next January. The sooner that speculation is turned into certainty the better for all parties.

## Dyestuffs Licences for July

The following statement relating to applications for licences under the Dyestuffs (Import Regulation) Act, 1920, made during July, has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee:—The total number of applications received during the month was 698, of which 575 were from merchants or importers. To these should be added 5 cases outstanding on June 30, 1930, making a total for the month of 703. These were dealt with as follows:—Granted, 663 (of which 637 were dealt with within 7 days of receipt); referred to British makers of similar products, 27 (of which 23 were dealt with within 7 days of receipt); outstanding on July 31, 13. Of the total of 703 applications received, 660, or 94 per cent., were dealt with within 7 days of receipt.

## German Dye Trust

The I.G. Farbenindustrie reports for the second quarter of the year that there has been no general decline in its business in consequence of trade depression. Its dyestuffs trade continues steady, though the developments in other countries that formerly depended on Germany for supplies has inevitably restricted its export business. The range of I.G. dyestuffs and intermediates continues to extend, and 1930 is expected to provide better trade than last year.

Fashion tendencies appear to favour a return to darker colours. In accordance with the rationalising process, the selling organisations established at Frankfort, Hoechst, Leverkusen, and Ludwigshafen, are to be centralised in a new administration building in Frankfort. This country, which was formerly a very large customer, has for years past drawn mainly on its own productions, and the reopening of the English market to unrestricted competition would no doubt result in renewed interest in our home trade on the part of the German makers.

## Rash from Dyes

A case recently decided at the Middlesex Guildhall draws attention to the importance of the purity of dyestuffs used in garments in contact with the skin. Dr. Leggett, of London, described as a radiologist, bought from a London firm a couple of hats, and in each case, it was alleged, a rash was caused by the dyed hat lining coming into contact with his forehead. The trouble was diagnosed as dermatitis. The plaintiff was awarded £10 damages.

## American Colour Fashions

In the 1930 Fall Hosiery Card issued by the U.S.A. Textile Colour Card Association, twelve colours are shown, including four shades repeated from past seasons because of their continued popularity. The new colours are Caresse, Oak Rose, Acajou, Nightingale, Mauve Beige, Promenade, Brownleaf and Bahama. The four repeated shades are Sable, Rendez-Vous, Duskee and Beige Clair. In addition to the above colours, the following eighteen shades, appearing in the standard hosiery cards, are recommended as being worthy of continued promotion for the 1930 fall and winter seasons:—Champagne, Grain, Gunmetal, Lt. Gunmetal, Afternoon, Allure, Almora, Breeze, Crystal Beige, Suntan, Basque Brown, Blond Doré, Florida, Muscadine, Plage, Rosador, Sunbask, Sunbrown.

## Master of Science in Textiles

The unprecedented growth of the rayon industry has resulted in an imperative demand for men who have had specialised training in various phases of chemistry and dyeing. In an effort to meet this demand the Textile Chemistry and Dyeing Division of the Textile School of Clemson College (S.C.) has developed a number of special courses for graduate students. These courses will be under the direct supervision of the Director of the Textile School, and Dr. C. E. Mullin, Head of the Textile Chemistry and Dyeing Division. They will be assisted by other members of the textile faculty, all of whom have had considerable practical mill work in addition to their training in theory.

The new courses include special aspects of textile research, spinning, weaving, testing and dyeing; advanced work in synthetic organic chemistry, particularly in relation to organic dyestuffs and textile chemicals; advanced analytic organic chemistry, covering organic analysis as applied to textile and related products; advanced work in technical writing and the study of technical literature; technology of textile fibres; advanced dyeing; textile printing; chemistry of starch; textile finishing; and the manufacture and properties of the artificial fibres. Upon the satisfactory completion of the required graduate work, the student becomes eligible for the degree of Master of Science in Textiles.

## Dyeing Cotton and Rayon Materials in Black Shades

By A. J. Hall, B.Sc., F.I.C., F.T.I.

*At a time when black seems to be returning to fashion, the author's contribution on the dyeing of cotton and rayon materials in black shades is appropriate. The second instalment of the article will appear in our September Dyestuffs Supplement.*

Up to the outbreak of war in 1914 the dyeing of all kinds of materials in black shades formed an important part of the work of the dyer, because black was everywhere regarded as a fashionable and standard colour. This demand for black fabric existed not only in England, but also broad and in the Far East. Since that time fashion has changed completely and bright colours have almost completely ousted black for many fabrics. At the same time, however, there remains a persistent yet smaller market for black fabric, whether it be of cotton or rayon, and in supplying this market there has been considerable progress among dyers and dyemakers. In spite of the appeal of colour, the dyer has not been allowed to forget how to dye a good fast black and has even been obliged to improve his methods, particularly as a result of the use of various types of rayon in fabrics which formerly consisted entirely of cotton.

Apart from Aniline Black, which is generally regarded as the most satisfactory of all black shades, the dyes used are chiefly drawn from the Direct, Sulphur, Vat, and Naphthol AS classes. These differ considerably as regards their fastness to light and washing, and the actual choice of dye must depend on the price allowed for dyeing and the use to which the material is likely to be put. The direct blacks have the least fastness; sulphur blacks are better and the vat and Naphthol AS dyes the highest degree of all-round fastness.

### Black Dyeing Processes

The peculiar influence of the market on the black dyeing processes employed is seen in the fact that America does but a comparatively small amount of Aniline Black dyeing as compared with that carried on in England; America's export trade in dyed fabrics to India and China is quite small, whilst it is to these countries that England sends large quantities of black material. The result is that American dyers are able to satisfy their home market with sulphur-dyed fabrics, and they therefore take but little interest in Aniline Black dyeing. On the other hand, English dyers are not able to satisfy the Far East with sulphur black, but must produce the highest grades of Aniline Black.

Direct black dyes have but a moderate degree of fastness and cannot be used on materials which must withstand repeated washing or exposure to light. In certain instances, however, their fastness to both of these influences can be much improved by an after-treatment with such substances as formaldehyde, copper sulphate, and sodium bichromate. An alternative method for securing increased fastness consists of diazotising the direct black dye on the fibre and then coupling it with a naphthol such as beta-naphthol, or an amine such as meta-tolulene diamine; it is obvious that for this process the dye must contain a diazotisable amino group. A distinct advantage of direct black dyes is that they have no adverse effect on the cotton or rayon fibres to which they are applied; they do not produce tendering at the time of their application or during subsequent storage.

Sulphur blacks are the most important of all the known sulphur colours, and are very largely used, in spite of certain disadvantages attending them. They can be applied satisfactorily to cotton and cellulose rayons (viscose and cuprammonium rayons) but have no affinity for cellulose acetate rayon. Because it is necessary to dye with an alkaline bath special precautions are required in dyeing materials containing cellulose acetate rayon—the one type of rayon which is readily saponified by alkalis.

### Cotton Goods

In the dyeing of cotton goods with sulphur dyes, which contain no artificial fibres, the conditions employed are those which are most economical as regards consumption of colour—it is not generally necessary to take any special precautions to preserve the handle or lustre of the cotton. Wherever possible, continuous methods of dyeing are employed, that is, long runs of fabric are led through two or more standing baths of the dye liquor and then immediately washed free from loose colour and dried. Certain difficulties have arisen in connection with this process in that the baths are liable

to gradual decomposition, and that it is not always easy to obtain deep well-penetrated dyeings, but the process is a cheap one and allows a large output—very much larger than is possible with the same amount of labour in dyeing on jigs. On the other hand there is less trouble with bronzy selvedges. If dyeing is carried out on a jig, then it is desirable to use a standing bath since sulphur black dyes do not exhaust well. Unfortunately, in this process of dyeing it is sometimes found that the resulting shades are bronzy, this being due to deposition of loose oxidised dye on the surface of the fabric. Whilst this defect can be corrected by passing the dyed fabric through a weak warm bath of sodium sulphide and rinsing, it is preferable to avoid the defect by the addition of a small amount of glucose to the bath. Normally only sodium sulphide and a small amount of soda ash is employed to maintain the sulphur dye in solution so that on exposure to the air the reduced dye readily becomes oxidised to its insoluble form. The glucose, however, does not oxidise so quickly, and thus restricts the oxidising action of the air. There is no doubt but that sulphur black shades on cotton have a more pleasing appearance when glucose is employed in the dyeing.

Sulphur black dyeings are of good fastness to all influences except chlorine, and would therefore perhaps be more widely used if it were not for the fact that on storage the dyed material is liable to become tender. This is always a danger which must be taken into account since it may involve a dyer in serious financial claims long after the dyeing of the goods has been forgotten. A few recently discovered sulphur dyes such as the Indocarbons (manufactured from carbazole) suffer less from this defect than others, and moreover, have a greater fastness to chlorine, but nevertheless, these should be dyed with care. Numerous investigations have shown that the aftertendering of a sulphur black dyed fabric is due to gradual formation of sulphuric acid in the fabric, and this is a result of oxidation of an active form of sulphur which accompanies the absorbed dye. This oxidation is brought about by the oxygen in the air and is promoted by catalytic metals in the fabric. So far the only real preventive measures that can be taken are (1) the use of a sulphur black dye which shows the least susceptibility to promote after-tendering; (2) the use of sufficient sodium sulphide in the dye bath so as to retard the deposition of free sulphur in the fabric, and (3) impregnation of the dyed fabric with acid neutralising substances. It is found satisfactory to leave in the finished fabric such substances as sodium acetate, sodium carbonate, and borax. Also, if the dyed fabric is after-chromed with a mixture of acetic acid and sodium bichromate, not only is free sulphur partially oxidised, but a certain amount of chromium oxide is left in the fabric and this acts as an acid-neutralising substance.

### Cellulose Acetate Silk

Turning now to the dyeing of cotton goods containing cellulose acetate silk, it may at once be noted that either a solid shade is desired or the rayon must be left white. In dyeing a solid shade, the cheapest method consists of first hydrolysing the rayon by a short treatment with a hot dilute solution of caustic soda just sufficient to give it an affinity for sulphur black equal to that of the cotton, and then dyeing the fabric in the usual manner at a low temperature. Alternatively the fabric must be dyed under such conditions that the cotton alone is dyed with the sulphur dye and the white or slightly stained rayon then dyed with any other black dye which is applicable to cellulose acetate rayon. It is evident that if the rayon is thus to be left white, no saponification must occur during the dyeing of the cotton. To accomplish this it is best to make use of the Lodge and Evans process, which was formerly devised for dyeing cotton-wool union fabrics. In this, the dye bath is prepared in the usual manner, except that sodium sulphate is present and an amount of ammonium sulphate added so as to convert the sodium sulphide into ammonium sulphide thus:—



Then, if dyeing is carried out at about  $40-50^\circ\text{C}$ . the cotton

becomes coloured without the cellulose acetate rayon being affected.

Experiments in connection with the above process by the writer has shown that although it is assumed that ammonium sulphide is formed by double decomposition in the dye bath, the resulting dyeings are considerably inferior to those obtained similarly, but using ammonium sulphide itself instead of the mixture of sodium sulphide and ammonium sulphide. Unfortunately such a method is not practicable, since the bath has a very objectionable odour and the process is costly.

In using the above saponification process it is very desirable to avoid saponifying the cellulose acetate rayon to too great a degree otherwise the cotton in the dyed fabric has a very bare or thin appearance.

It is obvious that cotton fabrics containing viscose or cuprammonium rayons can be dyed in solid black shades very much in the same manner as the saponified fabric described above.

There is no advantage to be gained by dyeing materials consisting entirely of cellulose acetate rayon with sulphur black dyes after saponification since excellent dyes specially suited to this type of rayon are available. At the moment black dyes directly applicable to cellulose acetate rayon are not entirely satisfactory, but it is likely that these will soon be available. Meanwhile developed blacks are largely used, and they have excellent fastness properties. Among these dyes S.R.A. Black IV and Dispersol Diazo Black A are well known. The dyeing process is based on the fact that cellulose acetate rayon is able to extract from a water suspension a water-insoluble aromatic amine (S.R.A. Black IV and Dispersol Diazo Black are amines), and that this on diazotisation within the fibre is able to couple with a suitable naphthol to form a black insoluble pigment.

#### Application of Developed Blacks

In the application of a developed black to cellulose acetate rayon certain important facts must be remembered. Firstly, in the diazotisation process sufficient time must be allowed for the completion of this reaction. It appears that the diazo reaction proceeds more slowly in cellulose acetate than in cotton, and this can be readily understood when it is remembered that the aromatic amine within this rayon is

very much like the amine dissolved in an organic solvent such as benzene. Again, increased time must be allowed for the coupling of the diazotised amine with the naphthol. This coupling may also be assisted by employing a naphthol solution at a higher temperature than is usual in azo dyestuff formation; towards the end of the process there is no objection to raising the temperature to 40 or 50° C. It is essential to take these precautions if a fast black shade is to be obtained. Frequently faulty dyeings may be traced to incomplete diazotisation and coupling. When correctly produced, a developed black on cellulose acetate rayon is very fast both to light and soap, but if badly dyed the shade tends to turn redder with alkalis, and this is extremely undesirable.

The naphthol almost universally employed as a coupling component is  $\beta$ -oxynaphthoic acid. It gives a bluish black, and the production of this tone of black is assisted by coupling in slightly acid (acetic acid) conditions—glue is added to the coupling bath to maintain the difficultly soluble naphthol well dispersed.

Black vat dyes may be applied by the usual methods to cotton and viscose rayon, but not to cellulose acetate rayon. A black dye of the anthraquinone group is available for dyeing cellulose acetate, but the resulting shade is not so fast to light as the black obtained with a developed dye. For the dyeing of 100 per cent. viscose rayon fabric there is no advantage to be gained by using a vat dye instead of Aniline Black; this last named dye will receive attention later.

There is one interesting method for dyeing a black shade on cellulose acetate of reasonable fastness since it involves the use of logwood. It will be remembered that this natural dye is largely used for the production of the best black shades on real silk materials, but is seldom applied to cotton. When cellulose acetate rayon is immersed in a concentrated solution of logwood extract it readily absorbs this dye and becomes coloured dark brown. If the rayon be now treated at about 50° C. for half an hour in a 1 per cent. solution of sodium bichromate, the logwood becomes oxidised and combines with absorbed chromium to form within the fibres a fast black pigment. The resulting shade is quite pleasing, having a slightly greenish tone, but it is not so fast as a developed black shade to light. Nevertheless, the dyeing process is so simple that it has much to recommend it.

(To be concluded next month.)

## Basic Intermediates for Dyestuffs : No. XXXVII.—Some Oxy Derivatives of Anthracene and Anthraquinone

By "Consultant"

THE oxy derivatives of anthracene and anthraquinone are among the more important dyestuffs intermediates, and may be classified into the three groups below:—

#### Oxy-Anthracene derivatives.

1.  $\alpha$ -Oxyanthracene or  $\alpha$ -anthrol.
2.  $\beta$ -Oxyanthracene or  $\beta$ -anthrol.
3. 1 : 5-Dioxyanthracene or Rufol. m.p. 265° C.
4. 1 : 8-Dioxyanthracene or Chrysazol. m.p. 225° C.
5. 2 : 3-Dioxyanthracene. Decomp. 180° C. From hystazarin.

#### Oxy-Anthrone.

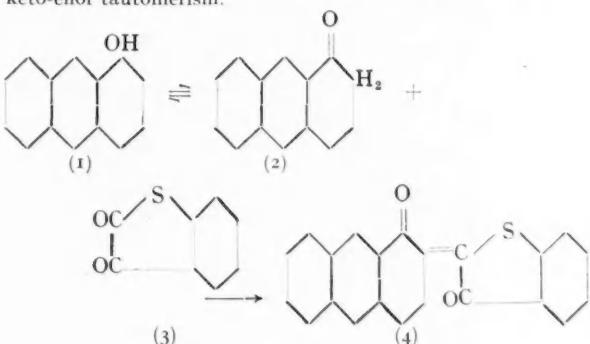
1.  $\alpha$ -Oxyanthrone. m.p. 221° C.
2. Dioxyanthrone, or anthrarobin.

#### Oxy-Anthraquinones.

1.  $\alpha$ -Oxyanthraquinone. Erythro-oxyanthraquinone. m.p. 190° C.
2.  $\beta$ -Oxyanthraquinone. m.p. 323° C.
3. 1 : 2-Dioxyanthraquinone. Alizarin. m.p. 290° C.
4. 1 : 3-Dioxyanthraquinone. Purpuroxanthin.
5. 1 : 4-Dioxyanthraquinone. Quinazarin.
6. 2 : 3-Dioxyanthraquinone. Hystazarin.
7. 1 : 5-Dioxyanthraquinone. Anthrarufin.
8. 1 : 8-Dioxyanthraquinone. Chrysazin.
9. 1 : 2 : 4-Trioxanthraquinone. Purpurin. m.p. 253° C.
10. 1 : 2 : 3-Trioxanthraquinone. Anthragallol. Occurs in Alizarin brown.
11. 1 : 2 : 7-Trioxanthraquinone. Anthrapurpurin.
12. 1 : 2 : 6-Trioxanthraquinone. Flavopurpurin.
13. 1 : 2 : 5-Trioxanthraquinone. Oxyanthrarufin.

14. 1 : 2 : 5 : 8-Tetraoxanthraquinone. Quinalizarin.
15. 1 : 2 : 3 : 5 : 6 : 7-Hexaoxyanthraquinone. Rufigalic acid.

Out of the list of anthracene derivatives given above for reference only a few are of importance as dyestuffs intermediates. Many are dyestuffs themselves—e.g., alizarin—and need no further chemical treatment. From the first group only one is of importance—namely,  $\alpha$ -anthrol. This compound (1) is able to condense with ketonic compounds such as thionaphthenequinone (3) to give compounds of the Thio-indigo dye class (4). It appears that the  $\alpha$ -anthrol behaves as a keto-compound in this type of reaction and is able to exhibit keto-enol tautomerism.



If the thionaphthenequinone is replaced by dibromisatin the so-called Alizarin Indigo is obtained, and in general this compound affords the means of manufacturing a series of vat dyes.

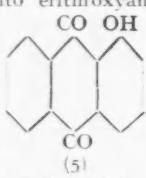
*α-Anthrol*

The preparation of  $\alpha$ -anthrol follows the usual process for the production of simple oxy compounds—namely, the alkaline fusion of the corresponding monosulphonic acid. The preparation of the anthraquinone- $\alpha$ -monosulphonic acid has already been described in this series (Part VII), and owing to the difficulty of sulphonating anthracene directly to a pure  $\alpha$ -monosulphonic acid, the corresponding anthraquinone sulphonic acid is used as a starting point for the preparation of  $\alpha$ -anthrol. The potassium salt of the anthraquinone monosulphonic acid (30 lb.) is heated with 20 per cent. ammonia solution (75 gall.) and commercial zinc dust ( $\frac{1}{2}$  cwt.). The progress of the reaction may be watched by the change in colour; at first the solution is intensely red, but as reduction takes place the mixture gradually becomes colourless and at this stage the mixture is filtered hot. On cooling, the potassium salt of  $\alpha$ -anthracene- $\alpha$ -monosulphonic acid separates in colourless leaves. The yield from this process (22 lb.) is mixed, while still moist, with caustic soda flakes (1 cwt.), and the whole fused in an iron kettle at 260–280° C. The replacement of the sulphonic group by the oxy group only takes half an hour, after which the partly cooled melt is rapidly dissolved in water and the solution acidified with hydrochloric acid. Dirty silver-coloured, minutely crystalline, masses of  $\alpha$ -anthrol, separate out. For condensation with keto-intermediates no further purification is necessary.

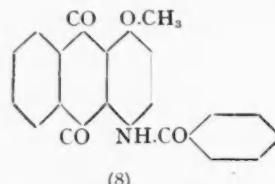
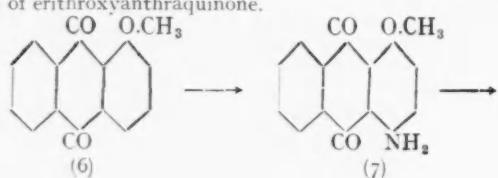
### ***Erythroxyanthraquinone***

***Erithroxyanthraquinone***

There are no oxyanthrones of any considerable importance to the dyestuffs industry, and of the oxyanthraquinones the first of importance is erthroxyanthraquinone, or  $\alpha$ -oxyanthraquinone, a bright yellow compound which has m.p. 190° C. The obvious method—namely, the treatment of the corresponding anthraquinone monosulphonic acid alkali, is the one which is used commercially for the preparation of this intermediate. Great caution must, however, be applied to the use of this method, since the amount of alkaline treatment necessary to replace the sulphonic group by the hydroxy group is far less than that associated with the benzene of naphthalene compounds. Drastic action, such as alkali fusion, results in the complete destruction of the anthracene molecule with the formation of a whole series of benzene derivatives. In actual practice, it is usual to convert anthraquinone monosulphonic acid into the erthroxyanthraquinone by stirring it with sixteen times its weight of 5 per cent. milk of lime in an autoclave at 190° C. for four hours. This will entirely convert it into erthroxyanthraquinone (5). The



mixture is cooled to 100° C. and the required intermediate obtained by neutralising the solution with hydrochloric acid, a little excess being used to ensure the precipitation taking place from a slightly acid solution. Pure erythroxyanthraquinone settles out as a yellow powder. It does not find extensive application by itself, but is converted to the corresponding methoxy compound (6). The Bayer patents recommend that this compound be prepared by the action of methyl alcohol and caustic soda on the correct anthraquinone monosulphonic acid sodium salt, hydrolysis and esterification taking place successively. There is much decomposition of the required product and the process is now obsolete, being replaced by the method involving the direct esterification of erythroxyanthraquinone.



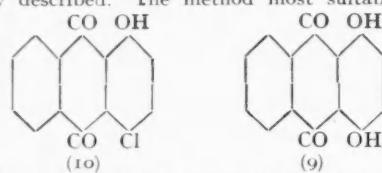
The process depends on the fact that when the methyl ester of an aromatic sulphonate acid is heated with the sodium salt of a stable oxy-compound the reaction

$R_1 \cdot SO_3 \cdot CH_3 + R_2 \cdot ONa = R_1 \cdot SO_3 \cdot Na + R_2 \cdot OCH_3$

takes place with resultant esterification of the hydroxyl group. In the case mentioned the sodium salt of the hydroxy-anthraquinone is heated with the methyl ester of toluene-4-sulphonic acid, the proportions being two parts of the latter to one of the former. The melt is conducted at  $180^\circ C$ . and the progress of the reaction can be watched by observing the separation of the methoxyanthraquinone as a pale yellow powder, which thickens the melt. The methoxyanthraquinone may be separated after the completion of the reaction by milling with spirit when the desired compound remains insoluble, and may be washed with water and dried. Its value to the dyestuffs manufacturer depends on the fact that it nitrates principally in the "4" position, and on reduction its nitro compound yields the amino compound (7). This on benzylation gives the well-known Algod Scarlet G. (8)

### *Quinazarin*

Among the dioxyanthraquinones only two are described as dyestuffs intermediates of the first rank, quinazarin and anthra-rufin. Quinazarin (9) is valued for its ability to condense through the hydroxyl groups with amines. The products so obtained are sulphonated to give acid wool colours. One such condensation with 4-toluidine gives rise to Alizarin Irisol; the condensation carried out on the two -OH groups is capable of producing Alizarin Cyanine Green. There are two processes that have been described for the production of quinazarin. The main process depends on the production of 1-hydroxy-4-chloroanthraquinone (10), a product which may be obtained by the chlorination of the erythroxyanthraquinone previously described. The method most suitable to large



scale production is that of Thomas and Scottish Dyes described in E.P. 209694. The principle involved is the direct chlorination of the erythroxyanthraquinone. The latter compound (1 cwt.) is dissolved with the aid of very gentle heat in commercial 98 per cent. sulphuric acid (5 cwt.) and the solution is allowed to flow down a small tower, up which a stream of chlorine is maintained, so that the temperature is kept at 70-75° C. The circulation of the acid is so arranged that about 5 lb. of chlorine is taken up per hour. The end point is judged by drawing a sample and pouring into water, filtering and testing the m.p. of the sample, when this has reached 180-181° C. the process is stopped and the product isolated by pouring into water. For conversion to the quinazarin this is not necessary, and the acid solution may be directly treated with boric acid (75 lb.) and subjected to the following treatment:—twelve hours' heating at 160° C. and eight hours at 180° C. The quinazarin is obtained from the solution by pouring into water and filtering off.

The alternative process is due to Bayer and has the disadvantage of employing parachlorphenol, a substance the preparation of which involves the preparation of the corresponding ortho derivative for which there is no large outlet. The parachlorphenol is condensed with phthalic anhydride in the presence of boric and sulphuric acids. The function of the boric acid is quite unknown; without it, however, only a minute yield of the desired product is obtainable. The phthalic anhydride (1 cwt.) is added to sulphuric acid (98 per cent., 5 cwt.) followed by boric acid ( $\frac{1}{2}$  cwt.) and parachlorphenol (44 lb.). The heat treatment for this reaction

mixture is three hours at 160° C. and such a period at 190-200° C. as will produce no further deepening in colour. The product is isolated by pouring into a small quantity of water

(5-10 gall.) and filtering off the paste that separates out. This is boiled with water, washed and dried, and constitutes commercial quinazolin.

## Dyes and Their Application: Recent Technical Progress

By L. J. Hooley

### Dispersion

A GOOD deal has been heard during the last two or three years on dispersing, solubilising and wetting agents, and the patent literature has described the production of a great variety of these substances as well as indicating avenues for their application. Dispersion is certainly an important consideration in certain aspects of dyeing, and, although the importance of dispersing agents for addition to soluble dyestuffs may sometimes be overrated, there is no doubt of their value for colours which are normally insoluble. This has been shown most clearly in the case of the acetate silk colours, but it is of equal importance with the vat and insoluble azo colours. A constant demand for the raising of the standard of fastness is calling vat colours into action for purposes for which they have not previously been used and for many of which the vat method of application is quite unsuitable, and for which the question of dispersion becomes even more important than ordinarily. In the following, some practical dispersion problems in the case of vat and other colours will be mentioned.

### Colour Pastes

Dyestuffs sold in the form of aqueous pastes, that is, dispersions of the dyestuff in water, sometimes cause trouble, because instead of remaining homogeneous they settle out. This is not of particular disadvantage if the proper condition can be restored by stirring. Sometimes, however, the colour either forms small lumps or masses at the bottom of the cask, which cannot be broken up again in this way; crusts of colour also sometimes form on the sides of the container. Although these troubles do not occur normally, they are liable to happen occasionally, no matter how carefully the paste may have been prepared. In this connection, something turns on the way in which the pastes are stored and the length of time they are in use, which may vary from dyehouse to dyehouse. A few methods have been suggested for avoiding the possibility of this trouble; one of these is the addition of a small quantity of a natural colloidal clay (B.P. 310,830, Harding Chemical Co., W. H. Clutterbuck). For the prevention of crusts and for improving dispersion, the I.G.F.A.-G. suggest the addition of esters of organic hydroxy acids, such as ethyl lactate (B.P. 318,176.)

Partly because of these difficulties and also because of transport costs, the use of powders instead of pastes is increasing. The difficulty in connection with these powders is that of obtaining them in as good a condition for dyeing and printing as the paste. Drying of the pastes, unless carried out with special precautions, destroys their dispersion, so that they do not give the same colour value on dyeing. The test shows up most clearly with the insoluble acetate silk colours. If these are improperly dried so that the products lose some of their solubility, it then becomes very difficult to get proper dyeing effects. Several processes have been recommended for making these dry powders. In many of these the drying is done in the presence of dispersing or solubilising agents. An interesting recent patent is B.P. 314,451 (I.G.F.A.-G.), where dyestuffs practically insoluble in water are incorporated with organic water soluble resinous and tenacious plastic materials. Thus 1-amino-4-hydroxy-anthraquinone is kneaded with water soluble casein and a small proportion of *p*-chlor-*m*-cresol and rolled in a friction mill until a uniform thin film is formed, which is dried in the air and then ground to powder—the product on dissolving in water gives practically a non-settling solution.

### Dyestuffs in Organic Liquids

Apart from the drying of pastes, the preparation of dyestuffs in organic liquid media requires the preservation of the degree of dispersion. With the spirit soluble colours this does not hold, as the process of solution provides a new dispersing effect and one which probably proceeds as far as the molecular

stage. The range of spirit soluble colours is, however, small and does not include light-fast members. In the use of insoluble azo colours and vat colours as pigments, particularly in media suitable for paints, inks and varnishes, proper incorporation of the colour often cannot be effected by grinding and the aqueous paste has to be converted to the varnish without intermediate drying, or to speak more accurately intermediate isolation in solid form. One recent method of effecting this can be seen in B.P. 318,937 (I.C.I., J. W. C. Crawford and G. E. Scharff), where water is removed by distillation in ternary azeotropic mixture. In an example, water-wet Scarlet Lake is mixed with methylated spirit and benzene and heated with stirring; a constant boiling mixture of water, spirit and benzene distils off and by adjusting the proportions of alcohol and benzene the colour can be left in alcohol, benzene or a mixture of both. The alcohol wet pigment disperses easily when introduced into solutions of cellulose esters, for example, nitro cellulose.

For an aqueous paste which has been once dried, a way of converting the dry paste to a physical form suitable for pigments is described in B.P. 320,359 (S.D.L., S. W. Dunwirth and J. Thomas). This consists of grinding the dry product with ether or other suitable low boiling solvents. Examples are given with vat and acetate silk dyestuffs, and the powders, as well as being suitable for pigments, give better results in dyeing and printing.

The amino alcohols, especially triethanolamine, have recently been recommended for a variety of purposes in the application of colour, and among them is the addition to printing pastes, especially with vat and sulphur colours (B.P. 324,315, I.G.F.A.-G.), among the amines mentioned in the patent are dipropanolamine and cyclohexylethanolamine.

### Printing

Dispersing agents have found considerable application in printing. In many cases it is even more important to have the colour in fine condition for printing than it is for dyeing—the reasons for this being obvious. Unsatisfactory physical condition may lead to many evils, such as uneven prints, prints lacking in depth, and speckiness, as well as in certain cases mechanical faults and damage to rollers. For this reason, even with pastes, specially prepared fine pastes are sometimes manufactured for the printer. However, with an increasing tendency to use powders, especially for export purposes, vat dyestuffs are being prepared in powder form in some cases even for printing.

### Use of Urea

A large number of substances have already been used for adding to printing pastes to improve dispersion and solubility. Recently Durand and Huguenin have patented the use of urea for printing mordant dyes. A colour such as Chromazurin E or Alizarine Viridine FF is mixed with urea, hot water and acid starch tragacanth thickening and then, after cooling, chromium acetate added; the cotton is then printed with the paste, dried, steamed and washed. Faster and more intense prints are obtained. The use of urea is not restricted to printing, but includes dyeing (B.P. 318,469).

Urea is also used by the S.C.I.B. for printing with vat dyestuffs, especially those requiring considerable quantities of glycerine to fix them. An example is given with Cibanone Orange 6R, a potash thickening prepared from water, maize starch, British gum, glycerine and potassium carbonate, being mixed with the colour, urea, sodium formaldehyde sulphoxylate and water (B.P. 318,979).

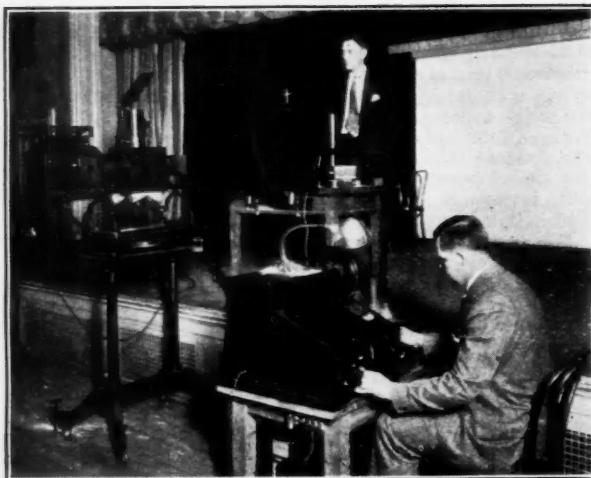
Another device for obtaining even prints is gelatinising the starch in the thickening in the presence of soaps or similar substances (B.P. 314,761, I.G.F.A.-G.). In one example, a weak starch is mixed with sodium oleate and gum arabic and gelatinised on the water-bath for 20-30 minutes. The process is stated to be especially suitable with vat dyestuffs.

**A New "Colorscope"**

By a New York Professor

THE physicist has again invaded the field of optics, and a function hitherto left entirely to the human eye is now to be performed by a laboratory apparatus of such simplicity of operation that it can be handled much as a modern radio set. The new "colorscope," built for the mechanical matching of two colours to a degree much closer than the eye could possibly achieve, was publicly demonstrated recently before members of the New York Electrical Society by their incoming President, Dr. H. H. Sheldon, Professor of Physics of Washington Square College, New York University, who with his assistant, Dr. W. A. Schneider, is responsible for this remarkable new instrument.

The principle of the "Colorscope" (so named by Dr. Sheldon in order to distinguish it from other forms of apparatus used



LABORATORY MODEL OF COLORSCOPE BEING DEMONSTRATED FOR FIRST TIME IN NEW YORK

only for colour analysis, etc.) is based on the photo-electric cell, already famous for its extensive applications in the control of sensitive apparatus, etc., by its reaction to light beams which make it give off infinitesimal electric currents capable of operating relays which in turn will start or stop machinery, operate graph needles, or perform other such laboratory and shop service. In this particular apparatus, two photo-electric cells (of any one of the sensitive materials, such as potassium hydride, calcium, selenium, thorium, etc.) are used, creating a "balanced" circuit under a normal light source. That is, they are connected to a galvanometer in such a manner that, when both receiving the same amount of light, they will hold the needle at zero. If one or the other receives more light, movement is produced from it, and the needle will deflect to the side registering the heaviest radiations.

A piece of the material, a sample of dye, or any standard colour to be matched, is inserted in one side of the apparatus, before one cell, and reflecting the light from a single light source into the cell. The needle is now brought to zero by a dial control similar to a radio tuning dial. A sample of another material of supposedly similar colour, or of another vat of dye, etc., is set before one cell in place of one of the standard samples. If there is a difference of shade, even though it be too slight for the human eye to detect, the galvanometer needle will slip off—if darker, toward the standard sample; if lighter, toward the test sample. A variation of three points either way will register a shade difference of enough importance to be considered faulty matching; although it has been found that a variation up to five or six is often undiscernible to the naked eye.

Weave and sheen effects, in the case of cloth, must be taken into consideration; as these create variations in readings which do not come from colour differences. The former is overcome by rotation, which welds the thread pattern into one solid mass of colour; and the latter can be counteracted

by the use of an integrating sphere, in which the light strikes on the cloth and is reflected in all directions, so that a concentration of light at any one point is eliminated.

This apparatus was first developed at the request of a large textile manufacturer; but as is often the case with scientific work done for a specific case, the potential applications seemed so much broader than originally expected that it was considered to be valuable for every industry using paints, dyes, coloured lights, or, in fact, any forms of colour.

**Dyestuffs Trade in Scotland**

JULY has been rather too broken up by holidays to form an accurate estimate of trade conditions. Probably, allowing for these, there is little change compared with last month, and the weather can probably be held partly responsible for some of the more pessimistic opinions. Dyestuffs sales, which are generally regarded as a fairly good index, do not show business to be so bad as it is sometimes made out.

Round Glasgow and district the holidays, which have already been mentioned, have been the outstanding feature. In the South the tweed trade are beginning with orders for next spring. Reports state that the greatest demand is for novel effects, the overseas trade especially being most attracted by the new designs. On the whole, buyers from both America and Europe are moving very cautiously at present.

With the Dyestuffs (Import Regulation) Act 1920, nearing the end of its normal period, the address of Sir Henry Sutcliffe was followed with very great interest as representing the official view of the Colour Users' Association. Perhaps the most interesting feature from the manufacturers' point of view, was the suggesting of a compromise on the question of further safeguarding. Sir Henry drew particular attention to the progress made during the past two years. This is a very important point and one which needs to be borne carefully in mind in considering the results obtained under the period of protection allowed by the Act. In a sense, it is only during the last two or three years that the industry has shown the progress which may be expected now that it has been established internally on sound lines. If the progress of the last two years can be maintained for two or three more, the future should then be faced with confidence.

**Spanish Commission on Colour Imports**

In view of a tendency which has arisen to interpret variously the provisions of the Royal Order of March 5 relative to the importation of colouring matters, the Government has issued a Royal Order which provides for the formation of a commission to study and co-ordinate the various opinions. This commission will function under the Ministry of National Economy and will be composed of the Director-General of Labour, the Chief of the Customs Division of the Direction-General of Commerce and Tariff Affairs, the President of the Barcelona Chamber of Commerce, representatives of manufacturers of Spanish colouring materials, as well as consumers.

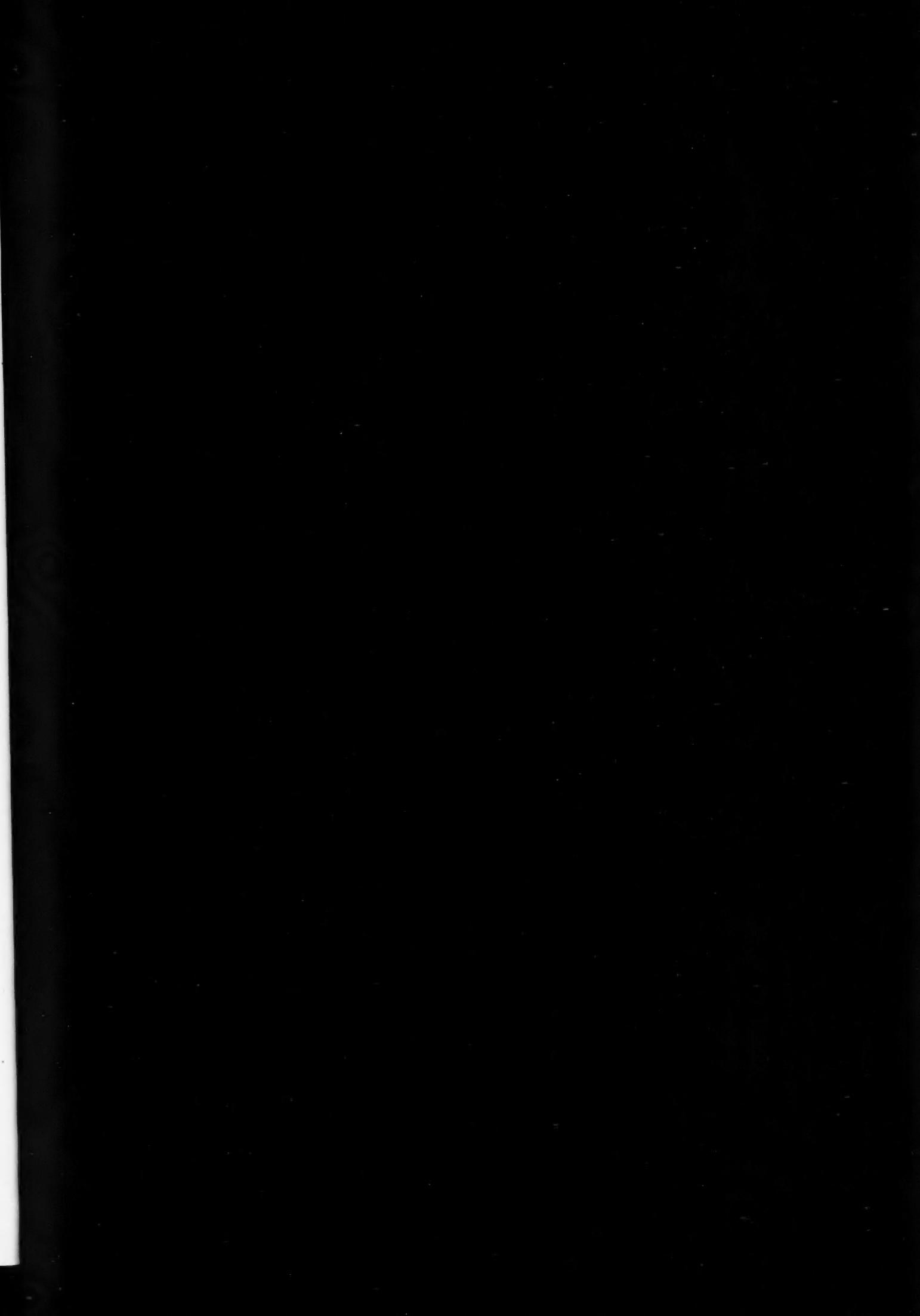
**Continental Trade Reports**

FROM Rome, it is reported that trade in industrial chemicals is slightly better, particularly in heavy chemicals and dyestuffs for the textile and tanning industries. Citric acid exports are noticeably less, while tartaric is slightly higher. Favourable conditions in the fertiliser trade are reflected by a 25 per cent. increase for the quarter in superphosphate output.

From Oslo, it is reported that the output of the electro-chemical industry is greatly increasing and there are no signs in the chemical trade of an industrial slowing down.

**Japanese Dyestuff Trade**

IMPORTS into Japan of dyestuffs decreased by 1,110,000 pounds, or 67 per cent. in volume, and 56 per cent. in value, during the first quarter of 1930, as compared with the same period of 1929. The domestic dyestuff industry, which is subsidised by the Japanese Government, is somewhat depressed, according to the recently published report of the Japan Dyestuff Manufacturing Co. of Osaka.



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# Dyestuffs Monthly Supplement

Published in the second issue of "The Chemical Age" each month

Communications relating to editorial matter for the Dyestuffs Monthly Supplement should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Advertisement matter, subscriptions, etc., should be sent to the Manager. The Supplement is devoted to the interests of both manufacturers and users of dyestuffs, and contributions will be welcomed.

## Dyestuffs Symposium at Bristol

Professor G. T. Morgan and his fellow officials of the Chemistry Section of the British Association are to be warmly congratulated on the success of the dyestuffs symposium organised during the meeting of the British Association at Bristol on Friday. The contributors to it were representative of almost every aspect of the industry—scientific, technological, commercial and national—and each was a recognised specialist in one branch or another and more or less an authority on the whole. With the exception of Sir Joseph Turner, these experts were all emphatically in favour of the continuance of the Dyestuffs Act in some form. They are not violently political people, who have taken up this position because of some rooted political theory in which they have been brought up. They are not diehards of the anti-Free-Trade or pro-Tariff types. They are mainly scientists, concerned about the future of the new British dyestuffs industry, chiefly for what its success or failure means to British chemistry and to chemical industry as a whole. A flourishing dyestuffs industry means a strong school of organic chemistry; it supplies a field for research chemists, technologists and chemical engineers; it guarantees—what the users themselves insisted on when they were suddenly cut off from their foreign supplies—an assured supply of home-produced dyes; it is in certain emergencies, which everyone hopes will never recur, of the highest national importance; beyond all this, it is closely linked with research work that may have medical results of great value. These are all substantial grounds that cannot be lightly disregarded, and those who desire to set them aside may reasonably be expected to produce considerations of equal weight.

## The Opposition to the Act

Where does the real opposition to the Act come from? Ignoring for the moment a certain fatalist mentality which has lived on a political theory that it regards almost as sacrilege even to discuss, there are first of all the merchanting community. Their attitude is at least consistent. Any restrictions on free imports are obviously restrictions on their own business, and logically and quite naturally they desire to get rid of them. This attitude is intelligible, although it has no relation to the considerations put forward at Bristol because it is not interested in a British school of chemistry. Then there are the users of dyestuffs. These are intimately associated with the industry, but their attitude is solely concerned with prices and with access to dyes, especially novelties, not produced in this country. Both points are important, but they were not the governing considerations even among the users when the Act was passed. What they insisted on then, as everyone else did, was a British source of supply which should never again leave them at the mercy of the foreign producer. Now they seem to argue, "Yes, that was very well during the war, but the war is over, and nothing like it is ever going to happen again." It is simply a return to the trustful pre-war mentality—which, with ample foreign supplies available, did not feel the need of a home industry, and refused to believe in the possibility of the arrest of such supplies. The dyestuff user has once more his world market to buy in. Why bother about guarantees against a danger

that has now disappeared? The best answer is that supplied by Mr. James Morton. He, as a 1 per cent. maker of dyes and a 99 per cent. user of dyes, is anxious to see the Act continued. The vital difference between him and the users who desire the discontinuance of the Act is that he has not forgotten his experiences as a user who found his sources of supply suddenly cut off, and that, having played a great part in creating a new British industry of great importance, he does not wish to see the results so hardly won lost to this country a second time. The one dissentient voice at Bristol was that of Sir Joseph Turner. But his position is quite unlike those referred to. As we understand his argument, the withdrawal of the help given by the Act will be such a challenge to the makers that they will resolve, out of their own resources, to make the future position secure. If the Act is continued, he seems to argue, they may take things easily and not make the necessary effort. It is an interesting argument, which is sound to the extent of not relying on the Act to replace initiative, but which scarcely balances the weighty arguments that the scientific, technological and commercial experts combined to produce at Bristol.

## New I.C.I. Colours

Imperial Chemical Industries announce the production of three new colours—Oil Yellow ATS, Chlorazol Union Navy 2B, and Chlorazol Union Black SB. Oil Yellow ATS is an addition to the I.C.I. range of oil soluble dyestuffs, and is commended to manufacturers of nitro-cellulose, oil and spirit varnishes by reason of its excellent solubility, coupled with its resistance to heat and light. The new Chlorazol colours are additions to the I.C.I. range of Union dyestuffs intended for the use of garment dyers. They give uniform solid shades on mixed materials consisting of wool, cotton, natural silk and artificial silks of the regenerated cellulose type. Navy 2B produces serviceable blue shades, and Black SB deep shades of a rich "bloomy" tone. The method of application is the same as that given for the other Union dyestuffs, and consists of entering the goods in a lukewarm dye-liquor, containing 20 to 30 per cent. of common salt on the weight of material, raising to 190°-200° F. in half an hour, and dyeing at that temperature for a further half-hour. If the cotton is required to be darker than the wool, the goods may be worked in the cooling dye-liquor for 20 minutes. A special feature of these two colours is that they are eminently suitable for employment in the standing bath. Their general fastness properties are described as "very good," and it is claimed that they meet the requirements of all garment dyers.

## German Royalties Action

Although the action taken in the United States courts to test the validity of the sale to the Chemical Foundation of the German patents seized during the war resulted in the confirmation of the sale by the Alien Property Custodian, certain aspects of the transaction are to be reopened shortly. These relate to accrued royalties under licences granted under the Trading with the Enemy Act, but the points at issue would appear to be matters of account rather than a challenge to the principle of the judgment in favour of the Chemical Foundation.

## The Present Position of the British Dyestuffs Industry

### Comprehensive Symposium at British Association Meeting

The Chemistry Section at the British Association meeting at Bristol devoted the whole of one Session on Friday, September 5, to a discussion on the present position of the British dyestuffs industry. The debate lasted over three hours and was sustained by some of the foremost authorities in the country, all but one favouring a renewal, in some form of the (Dyestuffs Import Regulation) Act.

PROFESSOR G. T. MORGAN, President of the Chemistry Section, was in the chair, and at the outset said the reason for having the discussion was that in the dyestuffs branch of chemical activity they had now come to a parting of the ways. The Dyestuffs Act became law in order to safeguard the dye-making industry of the country. The chemists who had carried out the practical work of our dye-making factories had done remarkably well in the short time at their disposal, but 10 years was a very short period in which to make up the leeway of 70 years. Our chemists had made highly meritorious progress in the manufacture of many of the fine colours, and especially in the vat series. There was, however, still room for further development in the technique of manufacture of intermediaries, and a few more years of Safeguarding would restrict considerably our importation of key intermediaries.

#### Professor A. G. Green

Professor A. G. Green, formerly of the British Dyestuffs Corporation, opened the discussion with a paper on "The Renaissance of the British Dyestuffs Industry." He recalled a survey of the industry which he had made at the British Association meeting in Glasgow in 1901, dealing with the relative progress of the coal tar industry in England and Germany during the preceding fifteen years. The picture painted on that occasion was a sad one. From prosperous and promising beginnings, founded on the work of Perkin, Hofmann, Nicholson, Caro, Martius, Witt and others, the British dyestuffs and organic chemical industry had slowly declined or had failed to develop, whilst during the same period the German industry had prospered greatly.

During the first twelve years or so of the present century the relative position of the British dyestuff industry did not change materially. Owing to the effective monopoly which the German dyestuff industry had established, with consequent lower working costs and the advantages accruing to the Germans as the inventors and introducers of most of the newer dyestuffs, it was scarcely possible for the English firms to improve their position, but they continued to manufacture a fair range of products, though largely from imported intermediates.

It must not be supposed, as a section of the public was apt to think, that in 1914 we had no dyestuff industry in this country. We had a dyestuff industry, small and struggling under great difficulties, but a portion of which at least was endeavouring to remedy the errors of the past, and this had formed an invaluable nucleus for the great extensions which were to come. We also possessed a number of young chemists who had received a sound specialised training in dyestuff and textile chemistry in the technical departments of the Leeds and Manchester universities. There was available, in fact, both technical experience and scientific knowledge, which could be, and was, drawn upon later.

After references to the specific attainments of various firms in the production of dyestuffs, drugs, explosives and dichlor-diethylsulphide (mustard gas) during the early part of the war period, Professor Green went on to discuss the steps taken in conjunction with the Government to consolidate and enlarge the dyestuffs industry. As early as September, 1914, he said, Lord Moulton, with Lord Haldane, had initiated steps with a view to consolidating and enlarging under Government auspices and assistance the entire British dyestuff industry. An essential feature of the scheme was the appropriation of large sums for research. Unfortunately, however, the scheme was destined in great measure to go astray. Deprived of the personal guidance of Lord Moulton, who became engaged in the more urgent necessities of the Munitions Department, the development of the scheme passed into the hands of politicians and incompetent administrators and took a course opposed by all those best qualified to judge. A new company—British Dyes, Ltd.—was brought into existence in March, 1915, having an authorised share capital of £2,000,000, with a debenture loan from the Government of £1,500,000 at 4 per

cent. interest, with the right to nominate two directors. Only one existing firm (Read, Holliday and Sons) was absorbed, and the new factories which were subsequently erected at Huddersfield at enormous expense showed little immediate results so far as the production of dyestuffs was concerned. Repeated changes in the Board of Directors brought no improvement, and the position continued with little alteration until the end of the war.

Faced with this unsatisfactory result, the Government in 1919 endeavoured to link up their creation with a more virile organisation. Levinstein, Ltd., in view of their war work, had strong claims to be treated on a similar footing with Read, Holliday and Sons, more particularly in regard to the endowment of their research work, the extension of which was essential in order to place the industry on a safe footing and prevent a return to the former conditions of foreign dependence. About one million pounds had been paid by Levinstein, Ltd., during the war in excess profits duty, and a powerful plea was extended that this should be returned and utilised for research and development purposes. In this situation the Government saw an opportunity of getting out of their difficulties with British Dyes, Ltd., and at the same time taking an important step towards the unification and consolidation of the dyestuff industry. Therefore, in July, 1919, there was promoted the British Dyestuffs Corporation, which effected a merger of British Dyes, Ltd., and Levinstein, Ltd. Of the authorised share capital of £10,000,000 the Government subscribed £1,700,000 (half in ordinary and half in preference shares) in substitution for the debenture loan to British Dyes to which they were committed, whilst five million £1 shares were offered to the public. This merger placed under a single control 75 per cent. of the dyestuff producing capacity of the country, and its formation was largely dependent on the promised passage by the Government of a Bill limiting for a period the importation of foreign competitive dyestuffs. This legislation, the Dyestuffs (Import Regulation) Act, had come into force in January, 1921, and on the whole it had worked satisfactorily. It had afforded to the dyestuff industry a fairly sheltered position during the period of its renaissance, and this without imposing any injury upon the colour-using trades, although many incorrect statements had been made to the contrary. Unfortunately, through unsatisfactory internal conditions, and the existence in the country of large stocks of "reparation" dyestuffs, full advantage could not at once be taken of the opportunity, and much valuable time and effort was lost.

#### Difficulties of the B.D.C.

The period which followed the formation of the British Dyestuffs Corporation was one of great difficulty and stress. The initial inflation which succeeded the conclusion of peace soon gave place to great depression in the textile trades, while at the same time the constitution of the company, to which a large amount of money had been subscribed by the public, required the earning of immediate profits instead of laying the foundation of an important national industry. The Dyestuffs Act, moreover, had the disadvantage that it encouraged waste of effort by the dissipation of energy over too large a field and a too slavish imitation of foreign products with a view to preventing their importation. Research was discouraged, therefore, except upon lines which promised an immediate return.

The relations between the constituent members of the combine were wanting in harmony from the commencement, and this culminated two years later in the resignation of both the managing directors, the technical and the commercial, in whose hands the control of the business rested. Deprived thus of experienced technical leadership, and with the management left in the hands of non-technical business men and so-called administrators, the position of the Corporation passed from bad to worse. The war being over, and the Dyestuff Act passed, the Government and the public had

lost interest in the matter, and apparently ceased to regard the establishment of an organic chemical industry as of vital importance. Largely under the pressure of the banks, in whose hands a considerable part of the under-written shares still remained, the board concentrated upon a policy of reducing expenditure and accumulating liquid assets, thus sacrificing the main objects of the previous legislation. The first things to come under the hammer were the research establishments of the Corporation.

The research personnel of the combined firms (including those chemists engaged in the investigation of immediate manufacturing problems) had grown during and immediately after the war to a total of about 100. They formed as a whole an able body of men, who, as their experience increased, would certainly have become of great value to the British dyestuff industry. Excellent results had already been achieved in the splendid new laboratories at Blackley and Huddersfield. By repeated onslaughts, however, the numbers of the research staffs were continually reduced, and at the same time the morale of the remainder was seriously affected and individual effort discouraged. The culminating point was reached in 1923, when, in reply to a demand for a further large reduction of staff, Professor Green himself resigned his position as director of research of the Corporation. The disastrous and short-sighted policy which resulted in the loss to the dyestuff industry of many men of exceptional ability and special knowledge and the jettison of many important investigations, continued until the absorption of the Corporation by Imperial Chemical Industries, Ltd., in 1926. Under the protecting wing of this huge combine, guided by men having a knowledge and appreciation of the chemical industry and a realisation of the importance of research, the Corporation was once more able to pursue a progressive policy. Research staffs were again increased, manufacturing costs reduced, new dyestuffs brought out, and the whole position consolidated. He did not think that it would be an exaggeration to state, however—if expressible at all in terms of money—that the dispersal of the research organisation during the years 1921–1923 had resulted in a potential loss to the Corporation of one or two million pounds sterling.

Professor Green then made brief reference to the development of the firms independent of the B.D.C. and, later, the I.C.I., especially the Clayton Aniline Co., the British Alizarine Co., Williams, Ltd., L. B. Holliday and Co., Brotherton and Co. (who acquired the German-owned Mersey Chemical Works), Hickson and Partners, and others. Many of these had made great progress, and together they supplied about 25 per cent. of the total home requirements, in addition to a considerable export trade.

#### Post-War World Trade

The disposition of the world's dyestuffs trade had become completely different from what it was prior to the war. The total production of dyestuffs, and consequently, the world consumption, was about 195,000 tons. Germany, in place of producing 80 per cent. of the world's requirements, now supplied only 44 per cent.; she no longer had a world monopoly, and her manufacturing costs were largely increased. On the other hand, her principal factories, loosely united before the war in two Interessengemeinschaften, had since been welded into the I.G. Farbenindustrie, a corporation with £40,000,000 subscribed capital, paying a dividend of 12 per cent., under a single control and having greatly widened interests.

France had established a considerable dyestuffs industry, supplying about 8½ per cent. of the world's requirements; Italy a not inconsiderable one, supplying about 3½ per cent.; whilst the Swiss works, well established from early times, and now united, had largely increased their influence and rendered themselves independent of Germany for raw and intermediate materials. They also owned branch factories in England and America, and, excluding the production of these, supply about 5½ per cent. of the world's total. Even Japan, under Government assistance, produced a limited range of dyestuffs for home consumption and export to China. During the past two years the European producers outside England have entered into a convention to delimit their respective markets and prevent destructive competition.

The largest producer of dyestuffs, however, was America.

She had erected large works, and, with the assistance of European chemists, had not only rendered herself independent as to 90 per cent. of her domestic needs, but had also become a large exporter, a position favoured by an extensive protected home market, a favoured position as to raw materials and very large plants. In fact, last year America produced 22½ per cent. of the world's dyestuff requirements.

The British industry now produced about 25,000 tons of dyestuffs annually and supplied over 11 per cent. of the world's requirements—about half the production of the United States and about a quarter that of Germany. A promising feature was the fact that, whilst between the years 1925 and 1928 the respective shares of the world's trade absorbed by Germany, Switzerland and America had remained nearly constant, and that of France and Italy had declined a little, the British share had increased from 8·9 per cent. in 1925 to 9·5 per cent. in 1927 and 11·7 per cent. in 1928. In terms of absolute weight the increase was still more remarkable, the British production having increased from 14,600 tons in 1926 to about 24,900 tons in 1929.

#### Great Markets in the Past

The combined productive capacity of England, France, Germany, Switzerland, America and Italy to-day was probably at least twice the world's present requirements, but, as with other chemical products, the consumptive capacity of the world was capable of enormous expansion as larger populations came within the range of western civilisation and western necessities of life. The largest consuming country for dyestuffs was China, which used about 70 per cent. of the total indigo produced and nearly 1,000 tons of sulphur black annually. India was also a very large and expanding market, importing over 7½ thousand tons of total dyestuffs, consisting chiefly of sulphur black, alizarine colours and Congo Red. The Asiatic and Eastern markets constituted the main outlet for German dyestuffs. America, which during recent years had made considerable inroads into these markets, seemed to be losing ground, whilst the British exports were increasing.

Looking at the future prospects of the British dyestuffs industry and of the organic chemical industry of which it formed an inseparable part, Professor Green said among the prime necessities of further advance were an enterprising business organisation and an active and far-seeing research policy, not looking too closely for immediate results. Germany continued to maintain large research staffs and was still predominant over other nations in the prosecution of original research in the organic field. Without desiring to depreciate the valuable investigations at present conducted in our own chemical factories, he pointed out that the number of British patents in this field filed by the I.G. Farbenindustrie outnumbered those filed here by the manufacturers of all other nations, British and foreign, put together.

As to the fiscal question, Professor Green said he was strongly of opinion that the British industry had not yet reached a state in which further protection was unnecessary. It could not be doubted that the Dyestuffs Act had been of immense service. During the period of its operation this country had become self-supporting and was beginning to develop a not inconsiderable export trade in dyestuffs. It had established the manufacture here of large numbers of products not previously made in this country, and we had been able even to take a prominent lead in the discovery of new classes of colouring matters such as those used for dyeing artificial silk, and new members of the vat series. The industry gave employment to a large number of workpeople (estimated at about 11,000), and was essential to our national security in war and in peace. Last, but not least, it was a highly scientific industry encouraging the prosecution of scientific research in our universities and technical colleges, and thus reacted beneficially upon the entire chemical industry and upon many other national activities.

The present selling prices of dyestuffs, allowing for the changed value of money, were no higher, with the Dyestuffs Act operating, than the pre-war German prices; in some cases they were lower. Therefore, it could not be said with truth that our textile trades had suffered by its operation. He considered the reverse was the case. It seemed inconceivable that such an industry should be allowed to languish or to be exposed to the unrestrained competition of the protected manufacturers of America and the more firmly established

industry of Germany. A further period of State assistance seemed absolutely necessary to establish the position which had been won. Whether this assistance should be in the nature of importation under licence, or of an import duty, was open to discussion, but he felt certain that it would be little short of disastrous if, pending such a decision, our markets were left open to unrestricted imports. It appeared to him, therefore, to be essential that before its expiry in January next the Act should be extended in its present form for a further five years. As a permanent measure he was inclined to regard an import duty as preferable to restriction of imports by licensing, as the former would afford less interference with international trade, give more stimulus to improvements in manufacture, and avoid the dissipation of energy over too wide a field.

**Professor J. F. Thorpe**

Professor J. F. Thorpe (a member of the Dyestuffs Development Committee) declared that a setback to the organic chemical industry of this country would mean a serious blow to our research schools, and extinction of the industry would also mean their extinction. It must be remembered that organic chemistry was not only the basis of the dyestuffs industry and of the intermediate products related thereto, but it was also the foundation of the fine chemical industry in so far as it related to medicinal products and substances of therapeutic importance. Insulin and Thryoxaline (to mention only two of a large number of similar substances of extreme importance to human health and happiness) were organic compounds, and the discovery and development of this vast field awaited the skill of the organic research chemist. Moreover, the framework on which the whole structure of biochemistry would ultimately be built was organic chemistry, and it was clear that the chemistry of the life process would never be unravelled until the chemistry of the organic compounds concerned therewith had been established and the reactions of these substances defined. The same remark applied to agricultural chemistry.

The need, therefore, for well-established schools of organic chemical research, filled by men undergoing a thorough training in the methods of research, was evident, and was one of the essential scientific requirements of the nation. The schools of organic chemical research in this country were in a strong and flourishing condition; a steady stream of students passed through them, and there was no lack of employment, for the student nearly always found a post waiting for him when he had completed his course. This satisfactory state of affairs was mainly the outcome of the Dyestuffs Act.

**Mr. James Morton**

Mr. James Morton (Chairman of Scottish Dyes, Ltd., and a director of the British Dyestuffs Corporation) spoke both as a dye maker and a user of large quantities of dyes. Financially, he said, he was 1 per cent. a maker of dyes and 99 per cent. a user, and he supposed that as an individual he had the largest amount of capital of any person in this country invested in dyeing, inasmuch as his concerns were all private and mainly personally owned.

In 1913 he had not made a single one of the complex vat colours. In 1929 (apart from indigo) they made 2,659,000 lb. of these dyestuffs, including every one of the many necessary intermediates. Obviously, the dye makers had to begin by making and supplying colours the dye users knew and wanted, but the record in new dyestuffs made by British makers was far from negligible, and much better than was generally known. For example, of the 100 colours made and sold by Scottish Dyes, Ltd., 47 were new colours (32 being entirely new products and 15 new brands). Many of the former German colours were made by new synthetic routes, and the whole manufacture of the vat dyes was based on new and original methods. The company had to their credit the discovery of Caledon Jade Green, of which not only did they make now more bulk per annum than the total importation of vat colours before 1914, but larger quantities of it were made by Germany under licence from the company, and still larger quantities in America. Another original development on the part of the company had been the solubilising of the anthraquinone vat dyes, a feature which had long been wanted by the dyeing trade, as it made these valuable dyes available for wool and natural silk, and marked the beginning of a large new development in this section of dyes and dyeing.

Again, it was not until the chemists of the British Dyestuffs

Corporation had discovered new colouring elements that acted ideally on acetate silk that this fibre had come into its own and was able to take its place as one of the valuable factors in the textile trade. These dyestuffs were known as Ionamine and Duranal. They had since been imitated by chemists abroad, and had become the recognised media for the dyeing of acetate silk in all countries. To viscose silk the British Dyestuffs Corporation had also rendered signal service.

As one who had been in the textile trade for over 40 years and had been engaged in dye-making also since the autumn of 1914, he could say that in the past eight years there had been in the dye industry of this country more enterprise, more concentrated research and proved ability, more real achievement in the discovery of new materials and methods, than there had been in the whole of the textile industry of this country in the past eighty years. Since 1914, Scottish Dyes, Ltd., had investigated and produced in their laboratories numerous intermediates and 150 colours, mainly of the complex vat type, and had put on the market over 100 dyestuffs, 47 of which were new. They had erected laboratories, buildings and plant costing over a million pounds sterling, and had given employment in an entirely new and highly technical industry to about 1,200 men. This represented only a minor section of the British dye trade. He ventured to say that if the textile trade, at the proper season, had put a tithe of the concentrated research, ability and team work into its problems that had been given to the problems of the dye industry in recent years, Lancashire would not to-day be telling the tale of woe that one heard.

**Benefits to the Dye User**

Dealing with the question as to whether the dye industry had been nursed and raised at the expense of the textile industry, and whether the conditions had meant serious sacrifice and inconvenience to the users of dyes, he expressed the emphatic opinion that the balance was overwhelmingly on the benefit side to the user. Memories were very short, and nothing had caused him so much amazement, or amusement, as had the tone of patronage on the part of some of the users to-day towards the dye industry, contrasted with their suppliant gratitude of ten years ago.

There was a general impression that during subsequent years, especially since 1922, after reparations had stopped and the operations of the Dyestuffs Act had had their normal effect, the dyestuffs industry had been living on sufferance, under shelter of the Act, and at great sacrifice on the part of the users. What would have been the experience of users supposing the dye industry here had suddenly ceased, say, in 1922? The policy behind the German quotations during the past ten years—and he said this in no sense of grievance—was obviously to offer the colours that we manufactured here at a price that made our productions appear to be dear, whereas, for colours which we did not make, they had charged prices that were a long way above their economic value. Only as our varieties increased and as we added one by one those other colours did the price of each come down to something like the economic value. Some years ago a colour was being charged for by the German firms at 37s. 6d. per lb., until his firm had manufactured and issued it at 20s. The foreign quotation then became 15s. In another case the price of the foreign product had fallen from 12s. 3d. or more (the price before his company's product was issued) to 6s. 4d., the colour being one which was used in considerable bulk by users here.

It might be argued, he continued, by some users that there was a time when they could have procured from abroad colours common to the manufacture of both countries at prices much lower than the home quotations, and that the textile trade had suffered seriously in consequence. In regard to certain groups of colours he agreed that, for a period, a condition of this kind existed. While we were quoted high and quite uneconomical prices for colours that were not made here, users were quoted prices from abroad for certain dyes (the equivalents of which were made here), which prices, at the time, it would have been quite impossible for the home dye maker to have met. That was a considered policy on the part of the Germans, to upset the users here and to strike a blow at the operations of our Dyestuffs Act. It had caused unsettlement here and some bad feeling between dye users and makers, so much so that the continuance of the Dyestuffs

Act was for a time in jeopardy and was the subject of special discussions in Parliament. But the loyalty and goodwill of the majority of the users had prevailed, and the establishment, by the Licensing Committee, of a price factor had put an end to this overt act, with the result that dye makers here had faith to proceed, and, with bulk production and experience, were quoting prices which were fairly comparable with those of the foreign producers. During the past two years very few licences had had to be granted on the grounds of price.

If the lack of dyes, or high prices, had been a serious obstacle to the textile trade, this should be reflected in the returns of those branches of the trade dependent mainly on their dyes content, as distinct from the branches which were not dependent on dyes. The recent report of the Government cotton inquiry was most valuable and illuminating in this respect. It analysed and tabulated the export returns of the various sections of the textile trade. The sterling value of the dyed goods exported had increased from £26,569,000 in 1913 to £33,914,000 in 1929. There were decreases in the export of grey unbleached cloth and bleached cloth (in which no dyes were used), and increases in exports of printed goods (partially coloured) and dyed and dyed yarn goods (wholly coloured). Surely the last inference that could be drawn from the figures was that the dyes supply had been a deterring factor.

The Dyestuffs Act was not an easy Act to administer, and the Licensing Committee had had many difficult and intricate matters to adjust in the early days, but out of practically nothing at all they had gradually hammered out a working machine which had been invaluable to the whole dyeing and dye-making industries of this country, both technically and commercially. As a dye maker and a dye user he could testify to the fairness with which they had held the balance between the two interests. The statement that there was reticence on the part of foreign dye makers to give information and help was simply unfounded. The chemists of his companies paid periodical visits to the Continental makers, and they could not be given better attention.

Free trader though he had always been, and having fully in mind his heavy financial interests as a dye user, his strong recommendation was a renewal of the Act. Finally, he suggested a new provision for inclusion in any modified form of the Act—namely, that new colours from abroad which were practical equivalents of colours already made here should not be given licences for import. It had been very apparent that in many cases foreign makers had made new brands of existing types, with the distinct object of avoiding the provisions of the Act and getting a trade in them as new dyes. Where any new colour had a distinct new property that would be of value to our dye trade, however, we should certainly have it.

#### Sir William Pope

Sir William J. Pope, who also supported the plea for the continuation of restriction, said the demand for technical chemists for staffing the dye works was incidentally providing chemists for all our other large and growing chemical industries, as well as for our educational institutions. The country is now better provided than ever before with technical, industrial and academic chemists, and it could be stated confidently that, owing to the increased facilities for training and greater freedom of choice of a career, our chemists had never been so well equipped and so competent as at present.

#### Mr. T. Taylor

Mr. Tom Taylor (a member of the Dyestuffs Licensing Committee and of the Dyestuffs Development Committee), dealing with dyestuff lake colours, said the manufacture of lake pigments may be regarded as a useful and important ally to the dyestuffs industry, although through modern developments it was often difficult to define clearly when an organic coal tar colour ceased to be described correctly as a dyestuff and became a lake or pigment colour. Owing to the free importation period, it was some little time before the benefit of the Act was felt by the lake trade, but during the last seven years or so the prohibition upon the importation of lake colours had been practically complete. Importations had been infinitesimal, and this had enabled the British lake maker to establish an important trade to the advantage of the consuming industries. For instance, the production of British made printing ink was now some three times greater than it was

in 1913, and if we might judge from the balance sheets of the leading paint and linoleum companies, these trades had in no way suffered from their use of British made colours, apparently being exceptions to the severe depression existing in so many industries. It had been estimated, said Mr. Taylor, that the production of lakes in Great Britain was now over ten times that of 1913. This may be attributed almost entirely to the Act, and under no other form of safeguarding or protection could such progress have been made.

#### Mr. J. Rogers

Mr. J. Rogers said that to-day dyestuffs prices in this country are generally as favourable to the purchaser as in any country, but this state of affairs had been arrived at by anticipating future improvements in the cost of dyestuffs and intermediates. In the vat dyestuffs field the foreign firms had a lead, but this could be overtaken within the next five years.

#### Sir Joseph Turner

Sir Joseph Turner opposed renewal of the Act. In this country we had dyeworks able to manufacture double the quantity of dyestuffs needed here, and therefore, sufficient to deal with any emergency. There was evidence, also, from the most eminent of the dye makers of Germany, who had visited works here, that our works were equal to any of the most modern concerns in Germany. Therefore, said Sir Joseph Turner, the Dyestuffs Act had fulfilled its object in the matter of providing the necessary works. He was not blind to the fact that this country was alone in its need for having access to every dyestuff that could be manufactured; out of every 100 lb. of dyestuffs used here, 75 lb. went into goods which were exported. It was also essential, therefore, that we should be able to secure dyestuffs at a price equal to the lowest that was being accepted for those dyestuffs in any country in the world, but, although much progress had been made, that position had not yet been reached, and it could hardly be said that the Act had fulfilled its object in this latter respect. He contended that the Act should be abolished; our works were fully equipped and able to meet all contingencies, our chemists and engineers were equal to any in the world in scientific knowledge, and all that was needed was the proper guidance of these experts by thoroughly competent chiefs.

#### Mr. C. J. T. Cronshaw

Mr. C. J. T. Cronshaw alluded to our independence of foreign intermediates and said that whereas in 1913 approximately 300 tons of Beta Naphthol were imported, to-day none was imported. In the same way over 100 tons of Alpha-naphthylamine was imported in 1913 but none to-day. Approximately 300 tons of para-nitraniline were imported in 1913, but none to-day, and over 1,500 tons of aniline oil had also been eliminated. Similar progress had been made by British chemical plant manufacturers.

#### Professor M. W. Travers

Professor M. W. Travers, F.R.S. (Bristol) said on a recent visit to Frankfort he had seen the vast buildings for the central commercial organisation of the "I.G.," and had learned something of what institutions such as the new Frankfort University were doing for commerce. On the other hand, he knew that the modern universities in this country had not made contact with the commercial life of the day, and as a consequence, those who were pushing our dyes and other commercial products would be almost untrained, in the modern sense, and they would be pitted against the properly trained men whom Germany would be placing in the field.

#### Professor H. E. Armstrong

Professor H. E. Armstrong had sent a written communication which was read in his absence. He referred to his remarkably long and close association with "the chief actors in the great dyestuffs drama," beginning with the famous Hofmann, and including W. H. Perkin, the founder (1856) of the synthetic dyestuffs industry, Nicholson of Magenta fame and Crookes. A vast body of fact had been brought to light in the study of dyestuffs, and if we lost that industry we inevitably lost our place in chemistry. In his opinion, added Professor Armstrong, although still needing no little improvement in management, the industry had now been fully developed in this country, in a way to show that people like himself were fully justified in their estimate of the ability of the British chemists to carry on the industry here as effectively as

it could be carried on by anyone or anywhere. That the nation must grant it every necessary and proper measure of support was beyond question.

**Mr. W. J. Woolcock**

Mr. W. J. U. Woolcock (chairman of the Dyestuffs Development Committee), summing up the discussion, declared that after the statements that had been made in the course of this meeting, there could be no doubt whatever that an extension of the period of operation of the Act was required, above all, because the intermediates position needed strengthening and building up. The users and makers had worked very closely together, particularly during the past few years. They had been promised by the House of Commons some years ago that they should not be placed in an unduly disadvantageous competitive position. That promise had been

kept, and to-day, with the offer that had been made on the side of the makers, he could not conceive that anybody who was prepared to look fairly and squarely at the position could say that the period of the Act should not be extended. He could conceive of only one reason which would induce the user to continue to say, parrot-like, "ten years and no more"; that reason was that there had been elevated into a fetish an economic theory which was sound only in certain cases and in certain circumstances, and once one was driven to base a case on an economic theory and not on facts, that case must go by the board.

Apologies for absence were received from Professor H. E. Armstrong, Professor F. M. Rowe, Sir Thomas Robinson (chairman of the Dyestuffs Advisory Licensing Committee), and Sir Henry Sutcliffe Smith (chairman of the Colour Users' Association).

## Dyeing Cotton and Rayon Materials in Black Shades.—(II)

By A. J. Hall, B.Sc., F.I.C., F.T.I.

*The application of black dyes to Cellulose Acetate Rayon is dealt with by Mr. Hall in the concluding part of his article below. The first half appeared in our August Dyestuffs Supplement.*

IT is now convenient to turn to the application of oxidation blacks to cellulose acetate rayon, since these promise to be of considerable importance in the near future. In the first place, it may be noted that there is no difficulty in dyeing Aniline Black on fabrics consisting of cotton and viscose rayon or entirely of viscose rayon. Twill and satin fabrics of these types dyed with Aniline Black are largely used for linings, it being essential that the dye should be very fast. There is, however, one point in connection with the application of Aniline Black to a mixed cotton and viscose rayon fabric which deserves consideration. It is found that viscose rayon is able to absorb a larger proportion of the Aniline Black padding liquor than the cotton, so that the rayon dyes to a darker shade since the pigment formed within the fibres is proportional to the amount of padding liquor absorbed. Consequently in a cotton-rayon fabric there is always a tendency for the viscose rayon to be heavier than the cotton. If the cotton is intentionally dyed to its usual depth of shade then the viscose becomes too heavy and appears red in tone. On the other hand if the viscose rayon is dyed to a satisfactory depth then the cotton is weak, and is then more liable to green during wear. It is somewhat difficult to secure the same depth of shade on both fibres. The most successful result is obtained by thoroughly desizing and boiling-out the fabric before dyeing. The more thorough this preparation the greater the absorbent power of the cotton and the more nearly it approaches the rayon in depth of shade during the subsequent dyeing. Of course this difficulty does not arise in the dyeing of 100 per cent. viscose rayon fabrics. There is also the further interesting point that Aniline Black on viscose rayon appears to be faster to greening than a similar shade on cotton.

**Difficulty of Applying Aniline Black**

Hitherto the production of Aniline Black on cellulose acetate rayon has been unsatisfactory in spite of numerous efforts to overcome the difficulties inherent to the rayon. As indicated above, the process of dyeing Aniline Black consists of impregnating the textile material with a liquor containing aniline, a mineral acid (hydrochloric acid), an oxidising agent (sodium chlorate), and a catalyst (sodium ferrocyanide or copper sulphate), so that, on drying the fabric and then exposing it in a hot moist chamber, oxidation of the aniline to Aniline Black pigment occurs: this pigment, being insoluble, is retained by the fibres. Now cellulose acetate rayon is largely waterproof. At any rate, it absorbs much less liquor than does cotton or viscose rayon. So that the first difficulty in dyeing this type of rayon with Aniline Black is that it does not absorb sufficient of the padding liquor to give a deep black shade. A second difficulty is associated with the fact that oxidation does not appear to proceed readily within cellulose acetate.

When cellulose acetate rayon is dyed with prussiate Aniline Black it is scarcely possible to obtain more than a greenish yellow shade, even if it be dyed twice. To a large extent this unsatisfactory result is due to the salting out effect of the

comparatively large quantities of inorganic salts (sodium chlorate and sodium ferrocyanide) present in the padding liquor as required in this process; these salts appear to prevent absorption by the rayon of the liquor. Also it is likely that the catalyst—sodium ferrocyanide—finds difficulty in penetrating the rayon and therefore cannot function as it normally would in the case of cotton. A much better result is obtained by application of the copper Aniline Black process where the inorganic salts used are less in quantity. But even in this case the black is not usually satisfactory. Modification is necessary, and the necessary research has been mainly carried out by A. J. Hall and G. H. Ellis acting independently. These researches have brought to light various interesting and valuable facts.

It has long been known that a black shade equal to that obtained by oxidation of aniline can be produced by using para-amino-diphenylamine or mixtures of aniline and this diphenyl compound; the black is still termed Aniline Black, even if produced in this manner, because the black pigments produced by the two methods are probably almost identical. Now cellulose acetate rayon has a greater affinity for para-aminodiphenylamine than it has for aniline, and is certainly able to retain the aminodiphenylamine better than it can aniline in subsequent washing treatments. Hence the discovery of an alternative process of dyeing Aniline Black consists of first dyeing cellulose acetate rayon with para-aminodiphenylamine as with an S.R.A. dye in a jig or winch machine, and then treating in a separate bath with an oxidising agent.

Unfortunately para-aminodiphenylamine is expensive, being about eight times dearer than aniline, and this militates against its use. Further, the same process using a mixture of aniline and para-aminodiphenylamine does not appear to be successful. Ellis has, however, found that mixtures of aniline and para-aminodiphenylamine can be used in the continuous process of dyeing, providing swelling agents are employed to increase the absorptive capacity of the rayon. Such a process is covered in E.P. 311,435, in which equal parts of the amines are employed and a mixture of lactic acid, acetic acid, and methylated spirit serve both as swelling agents and solvents for the amines. It must be admitted, however, that this method is considerably dearer than the normal Aniline Black process using aniline itself.

**Cheaper Aniline**

With the idea of using a cheaper amine and yet one that is very easily oxidisable, Hall (E.P. 258,699) has investigated the possibilities of 2:4-diaminodiphenylamine. This is prepared by reduction of the condensation product of 2:4-dinitrochlorobenzene and aniline and should be produced at a lower cost than para-aminodiphenylamine. It is found that it can replace the para-aminodiphenylamine whether in two-bath or continuous methods with satisfaction and yield a good black. Yet here is a difficulty. This particular amine has not hitherto found much use in the colour industry, and is not therefore immediately available.

With a view to overcoming this difficulty Hall further discovered that the amine could be actually prepared within the rayon and then oxidised afterwards. In this process (E.P. 322,893) the rayon is dyed in a soap bath with a suspension of 2:4-dinitrodiphenylamine which is very cheap and available; the resulting shade of the rayon is bright greenish yellow. By further action of sodium hydrosulphite this absorbed nitro compound can be converted to the corresponding 2:4-diaminodiphenylamine so that oxidation to black can then follow. Alternatively, sodium hydrosulphite may be added to the dye bath used for applying 2:4-dinitrodiphenylamine, so that this is reduced at or about the time of its absorption by the rayon. This latter method is better in that it allows considerable saving of time and cost.

But perhaps the cheapest method for producing a black shade in cellulose acetate rayon is that more recently described by Hall (E.P. 321,034), in which the rayon is first dyed with  $\alpha$ -naphthylamine and then oxidised with nitrous acid.

The treatment is rapid and all the products used are cheap. Furthermore the resulting black shade is of excellent fastness.

It is therefore obvious that most of the difficulties of producing oxidation (Aniline Black) on cellulose acetate rayon have been removed. Further improvements of the processes now available will be mainly with the idea of cheapening the cost of dyeing.

The above considerations show how much effort has been devoted to the dyeing of black shades on cotton and rayon materials during the past few years. Even much greater has been the amount of research and investigation carried out some twenty or thirty years ago on this one shade—black. Fashion is ever changing, and no one would venture to deny the possibility that in the future the popularity of black may return. If so, then there is no reason to doubt but that by that time dyers will be ready to dye satisfactorily the fastest blacks on cotton and rayons.

## Dyes and Their Application: Recent Technical Progress

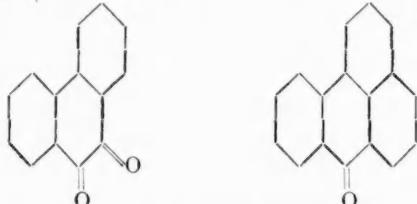
By L. J. Hooley

### Benzanthrone Dyes

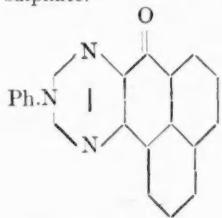
THE benzanthrone derivatives have been one of the most widely explored of dyestuff fields since the war. Some of the new products have found their way into use, but, as usual in dyestuff chemistry, these form only a small proportion of the total discovered. Dealing first with some benzanthrone intermediates, some of the recent advances will be found below.

### Intermediates

Benzanthrone is obtained from anthraquinone by a modified Skraup synthesis with glycerine. Structurally it should also be possible to carry out a similar synthesis from phenanthraquinone, but this has only recently been accomplished. A 58 per cent. yield of benzanthrone has been obtained, using glycerol and ferrous sulphate, the reaction proceeding in two stages, the first at 100° C. and the second at about 150° C. *Przemysl Chem.* 13, 33-5, 1929. J. S. Turski and R. Pragierowa.



The interesting series of analogues of hydrocarbons in which an N-phenyl-1:2:3-triazole ring takes the place of a benzene one has been extended to benzanthrone, 2-N-phenyl-1:2-naphtho-1:2:3-triazole, the analogue of anthraquinone, being condensed with glycerol in sulphuric acid containing ferrous sulphate.



This body can in turn be converted to dyes of violanthrone or isoviolanthrone type.

Methyl benzanthrones can be obtained from benzanthronyl thioethers. For example, 2-benzanthronyl thioethers of the formula  $S.CH_2.COR$  can be hydrolysed with alkali to give the corresponding 4-methylbenzanthrones (B.P. 322,745, I.G.F.A.-G.).

2-Cyanobenzanthrone can be obtained by the chlorination of 3-acetamidobenzanthrone, followed by hydrolysis, removal of the amino group and treatment with cuprous cyanide. (Swiss P. 122,904, Kalle and Co.).

Nitro groups in benzanthrone may be replaced by halogen

with acid halogenides, such as phosphorus pentachloride. In addition to the replacement, combination takes place at the keto group, and the resulting chloride has to be hydrolysed. In an example 13-nitrobenzanthrone is treated with phosphorus pentachloride in nitrobenzene at 95° C., the resulting chloride of 13-chlorobenzanthrone being then hydrolysed with water (D.R.P. Appln. J. 27,778, I.G.F.A.-G.).

### Halogenated Dibenzanthrone

Halogenated derivatives of the dibenzanthrone series have always been of importance, especially dichlorodibenzanthrone. Recently they have been attracting renewed attention.

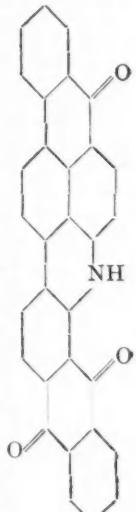
According to B.P. 294,486 (S.C.I.B.), the chlorination of dibenzanthrone in the presence of a high proportion of ferric chloride gives blue vat dyestuffs, which do not change in shade on moistening. In an example, chlorination is carried out in trichlorobenzene with ferric chloride at 140-150° C. Similar products can be obtained by treating 2:2-dibenzanthronyl with more than five times its weight of ferric chloride (B.P. 314,903, S.C.I.B.). Other highly chlorinated derivatives are prepared in B.P. 284,656 (S.C.I.B.) by treating already halogenated dibenzanthrones with chlorine and acetic acid at 130° C., the products being tetra- or pentachlorodibenzanthrones. A monohalogenated isodibenzanthrone is made in B.P. 315,276 (S.C.I.B.), by heating with chlorine or sulphuryl chloride. The product dyes in violet blue shades. Another one is also prepared in U.S.P. 1,704,983 (Grasselli Dyestuff Corp.) by condensing a monohalogenated 2:13-dibenzanthronyl. A large number of examples of bromination of dibenzanthrone and isodibenzanthrone derivatives are to be found in B.P. 325,525 (I.G.F.A.-G.).

### Complex Benzanthrone Derivatives

Recently there has been an extension in the direction of synthesising complex dyestuffs containing either benzanthrone, dibenzanthronyl or dibenzanthrone groups with anthraquinones, anthanthrones, etc. Among these may be mentioned first the comparatively simple benzanthronylamino-anthraquinones, one or two of which were already known before the war. These, on treatment, with condensing means such as alkali fusion, suffer further ring closure, giving black and grey vat dyestuffs (B.P. 307,732 and 316,156 I.G.F.A.-G.).

A little more complicated are the products of B.P. 300,432 (I.G.F.A.-G.), where tetrahalogenated 2:2-dibenzanthronyls are condensed with aminoanthraquinone. Thus the tetrabromo derivatives give a blue violet vat dye with 1-amino-4-methoxyanthraquinone.

B.P. 307,328 describes something similar, except that dibenzanthrones are used instead of dibenzanthronyls (I.G.F.A.-G.); while in B.P. 307,947 (I.G.F.A.-G.) aminodibenzanthrones are condensed with such complex residues as perylene, dibenzpyrenequinone and anthanthrone.



**Grey and Black Vat Dyes**

Black vat dyes are obtained comparatively readily from dibenzanthrone and dibenzanthrone derivatives, as these being already dark blue, little further is needed to give a grey. Many of the products thus obtained are, however, lacking in fastness. Some of the more recently described methods of producing blacks will be given.

Dibenzanthronyl sulphonic acids may be fused with alkali and then alkylated S.C.I.B. (B.P. 304,345). Alternatively, benzanthrone may be sulphonated, fused with alkali and alkylated (B.P. 297,002, S.C.I.B.).

Benzanthrone sulphonic acids have been fused with alkali and then treated with hydroxylamine to give greys, and substituted benzanthrones have been used in a similar manner (B.P. 280,652, 298,928, 298,931, B.D.C., Ltd., S. Thornley, A. Shepherdson and J. Baddiley). Aminebenzanthrone treated with alkali under mild conditions gives greenish-grey, green, grey and similar vat dyestuffs (B.P. 301,197, I.G.F.A.-G.).

**Benzanthrone Peri-dicarboxylic Acids**

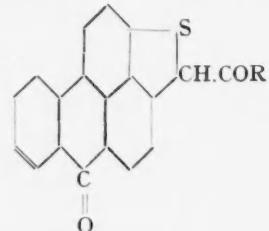
Within the last few years a new type of vat dye has been made by condensing peri-dicarboxylic acids with o-phenylenediamines, this reaction having been described for pyrene and perylene derivatives. It has now been extended to benzanthrone (B.P. 288,985, 308,651, I.G.F.A.-G.), 4-benzoyl-1:8-naphthoic or its anhydride used as a starting material for the necessary benzanthrone-peri-dicarboxylic acid into which it

is converted by aluminium chloride, being oncensed with an o-diamine to yield imidazole compounds. With o-phenylenediamine an orange vat dye is obtained, and with 1:2-naphthylendiamine a deep brown.

A rather remarkable reaction for the production of derivatives of the dibenzanthrone and isodibenzanthrone series consists in treating, for example, benzanthrone-13-phenoxy with o-chlorobenzoyl chloride, and then hydrolysing with sulphuric acid when isodibenzanthrone is obtained (D.R.P.J. 27,480, I.G.F.A.-G.).

**Indigoid Type Derivatives**

Compounds of the type shown in the formula below on



fusion with alkali are converted to derivatives probably of indigo type (B.P., 311,047, 316,172, I.G.F.A.-G.).

## **Dyestuffs Markets: The Month's Business in Review**

*From Our Own Correspondents*

**Lancashire**

No new facts have yet emerged that throw any light on the cause of the disastrous explosion at Castleford. All that is known is that the fire which preceded the explosion occurred in the acid-mixing shed, and not in the nitrating plant as was stated originally. In the acid-mixing shed, there should have been normally neither explosive nor even inflammable substances present. The first sign of anything wrong was the appearance of a cloud of dense brown nitrous fumes, which suddenly burst into flame; and the flames were followed by the terrific explosion before even the works fire brigade had had time to bring their engine into action. Fortunately, the works staff, aided by the fire brigades from Castleford, Leeds, and Pontefract, were able to prevent the fire from spreading to the benzol store, which was constructed of ferro-concrete according to the safety rules of the A.B.C.M. It is satisfactory to know that over £9,000 has already been subscribed for the relief of the hundreds of people rendered homeless by the disaster.

Sir Henry Sutcliffe Smith, at the recent annual general meeting of the Colour Users' Association, drew attention to the great increase in the productive capacity of the British dyestuffs industry, the annual output of which had grown from 9,000,000 lbs. in 1913 to nearly 56,000,000 lbs. in 1929. The most striking increase was in the manufacture of vat dyes, which had grown from nothing to 14,683,701 lbs. The pre-war production of dyestuffs in Great Britain amounted to 22 per cent. of the total consumption; in 1929 it exceeded 90 per cent. The average price of dyestuffs, in spite of the Dyestuffs Act of 1920, is less than double the pre-war price, whereas the price of benzol and toluol, doubtless owing to the increased demand for these bodies as motor fuels, is nearly three times what it was before the war.

The Colour Users decided that the Dyestuffs Act had achieved its purpose in assisting to establish the dyemaking industry in this country. Since too large a share of the burden of the Act had hitherto fallen on their shoulders, they declared that no further obstacle should be allowed to interfere with the unfettered importation of, at any rate, novelty and speciality dyewares from abroad. Lord Melchett and the Association of British Chemical Manufacturers, on the other hand, intend to press for a further period of protection on the lines of the present Act, on the grounds that, whilst a sound foundation has been laid, the removal of the present protection

would jeopardise the consummation of the work without any countervailing gain to the consumers.

The U.S.S.R., as part of their five-year plan for the resuscitation of Russian industries, have instituted a strong dyestuff manufacturing campaign. So far, the main articles being manufactured are fur dyes, basic and acid colours, and sulphur black.

October futures have tumbled to 6d. per lb., which is as low as in pre-war days, but the downward movement of the price of cotton is not bringing any more business to the mills. Buyers are holding off for a drop to 5d. per lb., or even less.

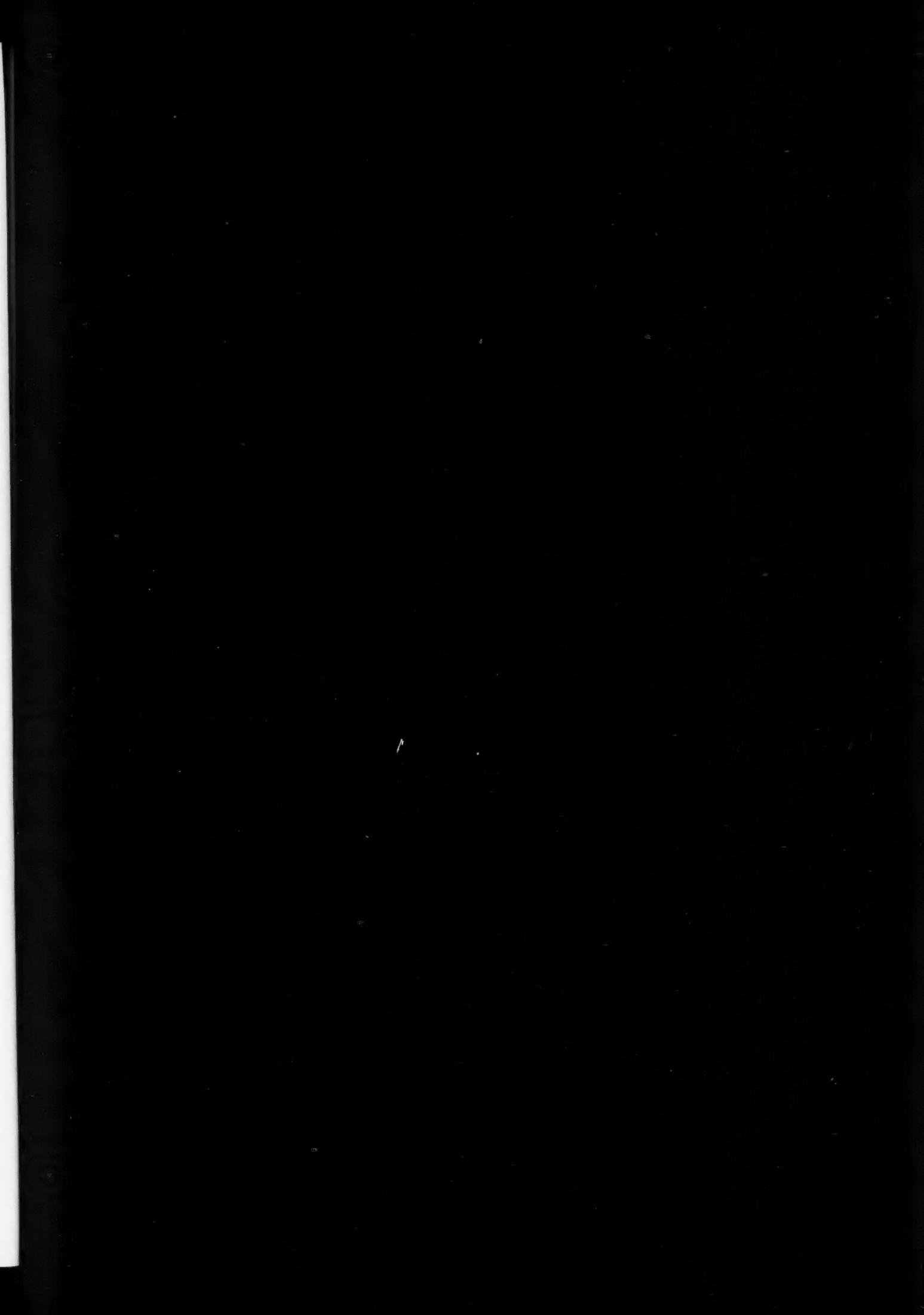
The report of the Bradford Dyers' Association is not calculated to relieve the gloom overhanging the textile industries, for the directors state that no profit has been made during the last half year.

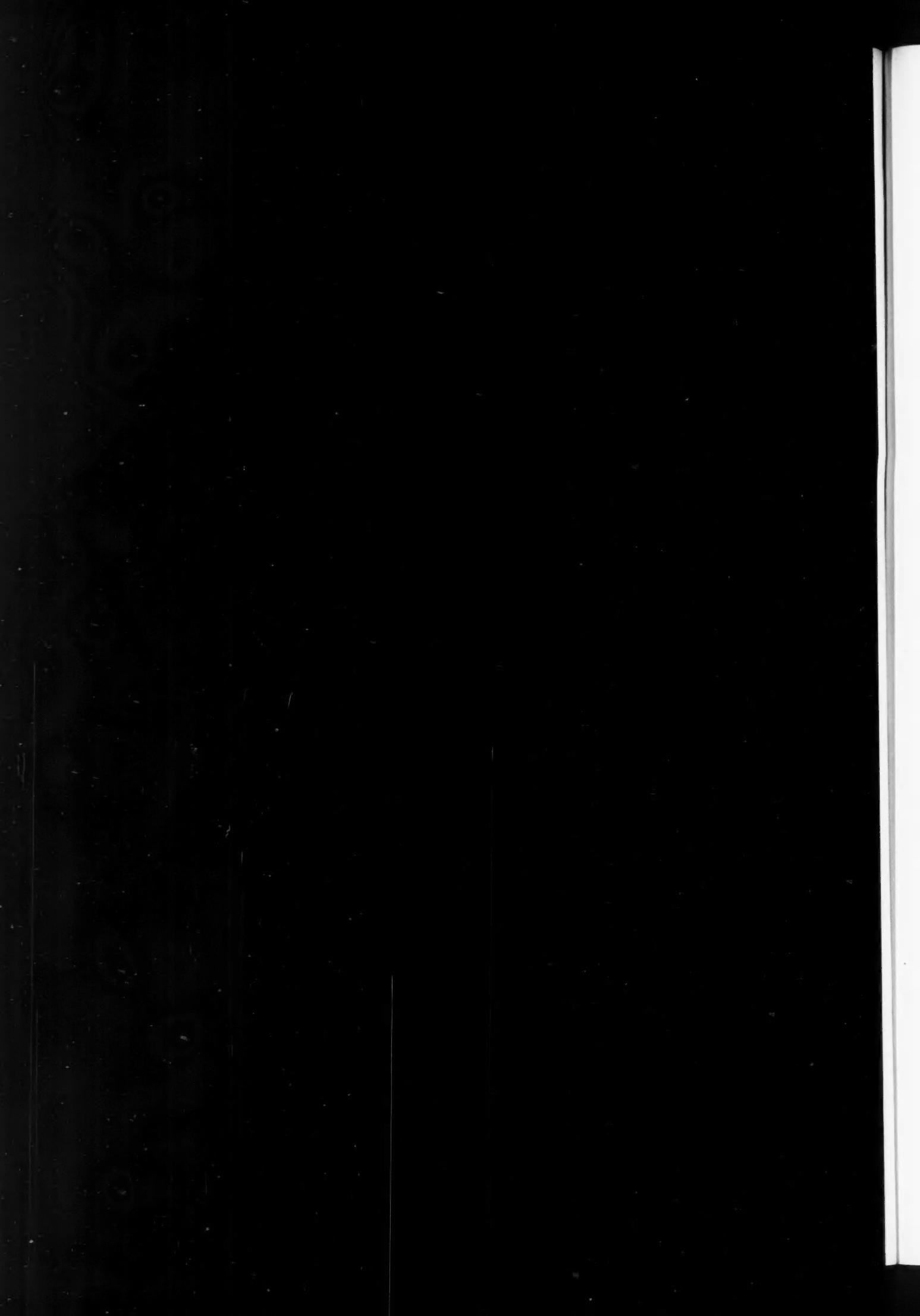
Great hopes have been dashed in Lancashire by the apparent failure of the expected financial assistance from the Bankers' Industrial Development Co. promised both by Mr. J. H. Thomas and by Mr. J. R. Clynes in public speeches to leaders of the cotton industry. But the proposed amalgamation of Egyptian or fine cotton spinners, for which Mr. Hammersley has been working, has apparently fallen to the ground owing to the rigid conditions under which alone the bankers are willing to finance any such scheme. The presidents both of the Master Cotton Spinners and of the Weavers' Amalgamation have expressed themselves very pessimistically at the set-back to this promising undertaking.

**Scotland**

With the publication by the Board of Trade of the report of the Development Committee, and with the discussion on the dyestuffs industry at the British Association Meeting at Bristol, this has been an interesting week as far as Scotland is concerned. The dyeworks at Grangemouth have good cause to be satisfied with such references as have been made to them. In the Report it is acknowledged that they rightly claim that in Caledon Blue RC they have the fastest of the fast blues.

At the British Association meeting, Mr. James Morton, speaking as one of the largest textile users, eulogised Scottish work and added that in the past eight years there had been in the dyestuff industry more enterprise, more research, more proved ability, more great achievement in the discovery of new materials and methods than in the whole of textile industry in the past eighty years.





# Dyestuffs Monthly Supplement

Published in the second issue of "The Chemical Age" each month

Communications relating to editorial matter for the Dyestuffs Monthly Supplement should be addressed to the Editor, THE CHEMICAL AGE, Bouvier House, 154, Fleet Street, London, E.C.4. Advertisement matter, subscriptions, etc., should be sent to the Manager. The Supplement is devoted to the interests of both manufacturers and users of dyestuffs, and contributions will be welcomed.

## The Manufacturers' Memorandum

We publish this week the memorandum which has just been issued by the Association of British Chemical Manufacturers, outlining the position and policy of the dyestuff manufacturers in the matter of the Dyestuffs (Import Regulation) Act. With its appearance it can be felt that all the necessary material is now available for a rapprochement of users and manufacturers, and the hammering out of a united policy. Subject to safeguards against unfair competition, the manufacturers ask for a prohibition of imports only where a British maker is prepared to supply an equivalent product at an equal price. The memorandum, which has a foreword by Dr. E. F. Armstrong, sets out the achievements of the dyestuffs industry during the period of operation of the Act. While based on the agreed facts in the Report of the Dyestuffs Industry Development Committee, it also takes into consideration the data for 1929, which have become available since the report was prepared, and, therefore, bring the account of the industry's progress more nearly up to date. The dyemakers pay a whole-hearted tribute to the "loyal co-operation of the colour users in the administration of the Act," and, in conclusion, appeal to them to assist in maintaining a modified form of the present safeguards without which there is nothing to gain and everything achieved during the past ten years to lose.

## "Dyes and Textiles in Britain: 1930"

The contributions to the recent symposium on the British dyestuffs industry at the British Association meetings at Bristol were all so good that each in itself would be worth reprinting. But if a choice had to be made, there is scarcely any with a stronger claim to reproduction than the paper by Dr. James Morton, which has reached us in the form of a beautifully printed pamphlet of 43 pages. This is not the first of Dr. Morton's contributions to what may be called the "literature" of the subject. His paper before the Royal Society of Arts will remain long after its delivery one of the most notable chapters of scientific and commercial achievement in the history of the industry during the most critical phase of its development. His Bristol paper, if not quite so pure a piece of history, has the advantage of being especially applicable to the problems of policy with which the industry is now confronted.

## The User's Point of View

The motif of the paper is clearly indicated in the author's foreword: "This paper was not prepared with any political object, nor from the point of view of any fiscal controversy. My life is mainly absorbed in industry and in the many problems which present-day conditions have brought up for solution. One of the problems facing the textile manufacturer, that of the future supply of dyes for his industry, was one of the most vital. The Dyestuffs Act was an interesting new method of dealing with what everyone regarded as a 'lapse' on the part of British industrialists of two or three generations ago, and an effort to make good that lapse with the least inconvenience to the trades involved. As the paper will show, the working of this Act has been intimately bound up with the most vital raw materials of the textile concerns which I control, and the fact that these concerns have not ceased to develop

successfully throughout the past ten years may make my findings of interest to others."

The findings certainly are of interest to everyone engaged in the industry, as the extracts given in this issue show. Two points, clearly brought out, are of special importance. The idea that the reopening of the British market to all foreign makers on equal terms would ensure cheap dyes is shown to be dangerously fallacious, in the sense that it may lead to the elimination of home competition in certain lines, with the certainty that the price to the home consumer will then be raised. The suggestion, too, that the slightly higher price of British dyes is damaging the export trade in finished goods is shown to be not supported by the facts, since the decrease is much more marked in uncoloured cloths than in partly and wholly coloured goods.

## Vulcafor Colours for Rubber

An Imperial Chemical Industries folder of more than usual interest contains samples of the fifteen Vulcafor colours produced for the dyeing of rubber. The effects of the colours as seen applied to ribbed-surface rubber is clear and brilliant. The complete range is made up of Vulcafor Brown Y, Claret R, Red B, Scarlet AS, Red RS, Orange AS, Orange Y, Yellow O, Yellow GS, Green 2Y, Green Y, Green LS, Green B, Blue G, Blue R, Blue AS and Violet RR.

Until comparatively recently, it is explained, the only practicable method of obtaining satisfactory colours in rubber was to use inorganic pigments. The colouring power of such pigments is usually low and a considerable quantity is required to produce medium to full shades. The Vulcafor colours are a specially selected range of organic colours which are for all practicable purposes insoluble in rubber, and which have been found particularly suitable for producing bright shades in rubber. Vulcafor colours soluble in rubber, such as Vulcafor Red IIS and Vulcafor Yellow IS, and water soluble colours for use in the colouring of latex are not included in the range shown.

For heat cures, the following mix was employed: Pale Crêpe, 100 parts; Zinc Oxide, 5 parts; Lithopone, 10 parts; Clay, 65 parts; Sulphur, 2 parts; Vulcafor VII,  $\frac{3}{8}$ ths parts. The cure was 10 minutes at  $141^{\circ}$  C. (40 lb. per sq. inch) in the press; slightly longer time is required for cures in open steam (cloth wrapped).

Where possible, a fast accelerator, such as Vulcafor VII, or VI, should be employed, although quite good results can be obtained with slower accelerators, such as Mercapto-benzothiazole or D.O.T.G. (Vulcafor XII). When D.P.G. (Vulcafor II) is employed, the yellow colour which it imparts to the rubber slightly modifies the resulting shade; for example, Vulcafor Orange AS will give a yellower orange closely resembling the generally accepted shade for inner tubes. In no case should accelerators such as Vulcafor Resin, which seriously discolour rubber, be employed with Vulcafor colours.

In general the effect of reclaims, dark substitute, and similar substances will be to dull the colour, and often entirely to change the shade; for example, a red or orange will become a brown. This effect can, of course, be largely overcome by the increased use of lithopone or titanium white. Common fillers, such as chalk, are without effect upon Vulcafor colours. The use of a limited amount of a white covering pigment, such as lithopone, is desirable to

give body and brilliance to the colour, but it should be kept in mind that a considerable increase in colour is necessitated.

For cold cures thin sheets of the following mix were cured by immersion in a 5 per cent. solution of sulphur chloride in carbon disulphide: Pale Crêpe, 100 parts; White Substitute, 50 parts; Lithopone, 10 parts; Chalk, 100 parts.

### Altex Union Colours

An interesting feature of another I.C.I. folder of Altex Union Colours is that in the eight shades shown the patterns consist of cotton, wool, silk, and acetate silk yarns. The eight colours are Yellow G, Red B, Brown G, Dark Green B, Light Green G, Saxe Blue R, Navy B, and Black DB. As to the method of application, Altex Union Colours are dissolved by pouring boiling water over them, stirring at the same time. They should be added to the dyebath through a fine sieve. Dyeing is conducted at a temperature of 180-185° F. with an addition of 10-40 per cent. Glauber's salt or common salt dependent upon the depth of shade; dyeing is continued for  $\frac{1}{2}$ - $\frac{3}{4}$  hour. The garment is then well washed and dried. No after-treatment or development is necessary.

All garment dyers, it is pointed out, will be familiar with the fact that the introduction of cellulose-acetate silk in the textile field has increased their difficulties. Whereas the dyeing of garments consisting only of cellulose-acetate is made possible by the use of Duranol and Dispersol Fast colours, garments which contain cellulose-acetate only in part create considerable difficulty. It many times happens that the presence of acetate silk is not suspected, consisting as it does sometimes as a lining or as a piece of trimming, and its presence is only manifest after the ordinary dyeing methods have been fulfilled. This brings about the necessity for a re-dyeing treatment with consequent loss of time. It was with a view to surmounting these difficulties that Imperial Chemical Industries introduced Altex Union Colours. These products, as the name implies, are capable of dyeing mixtures of all the various textile fibres—cotton, viscose, wool, pure silk and acetate silk. The card illustrates the first members of this new series of colours. The range is at present small, but will be extended as necessary to meet the requirements of the trade.

### A Group of General Colours

Seven new colours, being additions to existing I.C.I. ranges, are announced by Imperial Chemical Industries, namely, Caledon Orange 3RS Paste, Orange 5RS Paste, and Dark Blue WFS Paste, Durindone Pink FFS, Acronol Brilliant Blue 6GS, Duranol Violet RS Paste, and Chlorazol Black JHS. The effects shown on print, cotton piece and cotton, viscose, and woollen yarns are in several cases particularly delicate and attractive.

The two Caledon colours Orange 3RS Paste and Orange 5RS Paste represent important additions to the I.C.I. range of Caledon colours. They are suitable for dyeing all classes of cotton goods and viscose either in machines or in the open beck. Their excellent fastness to light and level-dyeing properties render them of particular interest to the casement curtain trade. Caledon Oranges 3RS and 5RS are dyed at 120° F. according to the particulars set out in the pattern card on Caledon colours. In printing, the products are applied in the usual manner, using either the Potash-Rongalite or Caustic soda-Rongalite methods.

Caledon Dark Blue WFS Paste, another new Caledon colour, is suitable for the dyeing of all classes of cotton materials either in machines or in the open beck. A special feature of this new product is the fastness of the shade to moisture.

Acronol Brilliant Blue 6GS is a new addition to the I.C.I. range of basic dyestuffs. Dyed on cotton mordanted with

tannin antimony, brilliant greenish blue shades fast to washing are produced, whilst in combination with Auramine OS it gives very brilliant yellowish-greens. On silk bright greenish-blue shades fast to water are obtained. In addition this colour is suitable for dyeing wool, artificial silk, straw, tagal and wood chip. Calico printers will find this colour suitable for the direct printing of cotton, silk and acetate silk by the customary methods of application of basic dyes.

Duranol Violet RS Paste, an addition to the Duranol colours, is applicable to all types of cellulose-acetate silk, and to mixed goods containing cellulose-acetate silk. Owing to its excellent affinity for the fibre it is of considerable interest as a base for the production of navy blue shades.

### Fastness to Kier Boiling

Durindone Pink FFS Paste, an addition to the I.C.I. range of vat dyestuffs, is suitable for application to all types of cotton material, giving shades of outstanding brilliancy and excellent fastness to washing, boiling, bleaching, etc. Its excellent fastness to kier boiling makes it of particular interest for the dyeing of cotton, hanks, warps or loose material which are subsequently bleached in the piece. It is suitable for calico printing, either for direct printing or for discharge effects by means of the Rongalite-Leucotropine process. Durindone Pink FFS is specially interesting for dyeing artificial silk, on account of its brilliancy of shade and fastness to washing combined with the valuable property of giving even shades on material of irregular quality. It is of interest for the dyeing of loose wool and slubbing giving shades of excellent fastness to milling and good fastness to light. Dyed on natural silk, the excellent fastness to degumming and good fastness to light will make this colour of particular interest to silk dyers. Durindone Pink FFS Paste is suitable for application to linen. For wool Durindone Pink FFS is reduced in concentrated solution as for cotton, but the dyebath is set with 3 per cent. Glue, 3 per cent. Ammonia (·880), 2 per cent. Hydrosulphite, at 140° F.

Chlorazol Black JHS, an addition to the I.C.I. direct cotton dyestuffs, belongs to the same class of colours as Chlorazol Black BHS but gives a much redder shade. It is capable of being developed with Beta Naphthol or Metaphenylene-diamine, giving full rich shades of very good fastness to washing, water and perspiration. It is applicable to all types of cotton materials, and on account of its very good solubility may be applied in the various types of circulating machines. Owing to its low affinity for animal fibres this colour is specially suitable for the dyeing of union materials where it is desired to dye the cotton and leave the wool or silk undyed. Dyed at temperatures below 120-130° F. it possesses very good affinity and covering power for the cotton and leaves the wool practically undyed. It may be applied in milling liquors for covering the cotton in wool-cotton union materials. It is suitable for dyeing viscose artificial silk and paper.

### Dyestuff Licences for September

The following statement relating to applications for licences under the Dyestuffs (Import Regulation) Act made during September has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee. The total number of applications received during the month was 507, of which 453 were from merchants or importers. To these should be added 28 cases outstanding on August 30, making a total for the month of 535. These were dealt with as follows: Granted, 512 (of which 481 were dealt with within seven days of receipt); referred to British makers of similar products, 17 (of which 15 were dealt with within seven days of receipt); outstanding on September 30, 6. Of the total of 535 applications received, 496 or 93 per cent. were dealt with within seven days.

## Has the British Dyestuffs Industry Helped the Users?

By James Morton, LL.D., F.R.S.E.

Our readers will be glad to have the full text of the terms in which Dr. James Morton discussed in his paper at the British Association the relations between the British dyestuffs industry and the interests of British users, and demonstrated, as a large user himself, the advantages that have been secured. The complete pamphlet is issued by the Association of British Chemical Manufacturers.

THE question of whether the dye industry has been raised at a sacrifice on the part of the users or otherwise cannot be stated in dogmatic terms by anyone, but my emphatic opinion is that the balance is overwhelmingly on the benefit side to the user. Memories are very short, and nothing strikes me with so much, shall I say amazement or amusement, as the tone of patronage on the part of some of the users to-day towards the dye industry compared with their suppliant gratitude, say, ten years ago. Such people forget that the industry was launched at a time of national starvation in dyes, and purely for the sake of the users. It was started on the assumption of goodwill and loyalty between parties to meet grave national emergency and future necessity. The attitude of some users and their Press to-day makes one think that they have forgotten all this, and that dyemakers are a kind of burdensome luxury they cannot afford and that they would rejoice to do without. Such persons would willingly send the new Hofmanns back to Berlin, or somewhere else, and revert to their old world of dreamland and the spoon-fed nursery that kept them so contented up to 1914.

Happily, however, that represents the attitude of a small minority only, though, like most minorities, they are apt to be the most vocal.

### German Price Policy

Now, in what I am about to say as to the policy behind the German quotations during the past ten years, I wish it to be understood that I say it in no spirit of grievance. It was quite an understandable business method of handling a situation, especially by a country that had been impoverished by years of the most expensive war in history. But what was that policy? It was obviously this—to offer the colours that we manufactured here at prices that made our productions look dear, while, in colours we did not make, to charge prices that were a long way above their economic value. Only as our varieties increased and we added one by one those other colours did the price of each come down to something like an economic value. That, to my mind, is the great, the incalculable service that the continued existence in this country after 1920 of a solid dye industry gave to the users.

In a paper some years ago I gave examples of a colour that was being charged by the German firms at 37s. 6d. a pound till our firm manufactured and issued it at 20s., to be immediately met by a quotation abroad of 15s. Another I gave that fell in a similar way from 12s. 3d. or more, before our product was issued, to 6s. 4d. a pound, a colour of very considerable bulk to users here.

### The Sphinx of the Dye Trade

These are comparatively old experiences now, but I want to give you one example that is of yesterday or to-day, and I should like your very careful attention to this story, for not only does it exemplify in a definite way my claim as to the price factor, but it may be regarded as a very correct epitome of the whole dyes question as it applies to this country to-day.

It refers to what used to be Algole, now known as Indanthrene Brown R and G. I think I might call this colour the Sphinx of the dye trade. We used it prior to 1914, and it had become a very vital colour. It was used for getting fast buff shades, and was specially valuable for fast-colour poplin suitings for the East. I had always in mind the early tackling of this colour, and my old friend, Sir Milton Sharpe, then chairman of the Bradford Dyers' Association, kept asking what hopes there might be for its production. We had peeped into its labyrinth, but, as there were so many other essential colours that offered easier solution, it was not till May of 1910 that I put one of our most advanced chemists on this problem, with his assistants. He was a Ph.D. of Zurich, and I thought we might land quickly at our goal. It was impossible to discover from the maze of patent literature what this Algole Brown really was, but by a happy hit we got at its constitution, and we were perhaps the only persons outside Germany for

years who knew what this product was. It is one of the complicated Benzoylamin Anthraquinones, and is formed from two different Anthraquinone derivatives, each of which requires seven different intermediate stages. Each of these has to be got to the exact degree of purity before they will combine to give the final product of the necessary tone and purity. As I have said, it proved the very demon. Although we knew its composition as early as 1919, it was 1922 before we actually produced it, and then of inferior quality. Early in 1924 we made a batch of 600 pounds that was really good in quality, but the yields were so low that the price was impossible. We kept pegging at it, and I can remember my visits to that particular laboratory for the Brown study week after week, month after month, till it grew into year after year, always getting reports of improvements in one phase that were knocked down by failures in others. You must remember the multiplicity of processes and materials that had to be just right at every stage. This continued, and although we were making some colour which we managed to use in our own dyeing works, it was not of a purity or at a price that I could allow to be put on the market as an equivalent of the German product. In 1927, when we began our collaboration with the chemists of the British Dyestuffs Corporation, we found that they also had been breaking their jaws on this bone. I think they would agree that they had not advanced so far as ourselves in the problem, but we discovered that at one stage they had obtained, by a different route, results that were ahead of ours, and, when we developed our process, with the addition of this rectification, we at last got our product perfect in tone, purity and yields.

### Competitive British Production

Such is the history of one colour. From 1919 to 1928, Dr. Beckett and his assistants had been engaged almost uninterruptedly on its solution. To the layman this will seem a long time for the perfecting of one colour. But the initiated will understand. Though Baeyer first made synthetic Indigo in his laboratory in 1880, it took till 1807, with the concentration and co-operation of the best chemists of the time in Germany, before the yields and other conditions could be brought to the stage that enabled it to be put on the market against the natural product; and chemists will agree that from the point of view of chemical constitution Indigo is simplicity itself in comparison with the Brown we are discussing. Moreover, our work, apart from the specific purpose, has had most valuable results, for from what seemed endless and hopeless investigations, although we did not jump into perfect Brown R, it was through these that we got many other compounds or derivatives, new and otherwise, which later formed the basis of the Scottish Dyes' Celatine series of colours for Acetate silk, now manufactured in a large way, and which we had patented just a few weeks later than the products of the British Dyestuffs Corporations' similar group.

### Cutting Out the Home Maker

Thus we were now ready to put our Brown R and G on the market and to replace the foreign import. What was the price to be? In the light of our newly acquired knowledge, we knew that, with the long experience the Germans had had and their bulk production, it was a colour that could not be very expensive to make. We knew that under such conditions a figure in the neighbourhood of 3s. per lb. would show a very ample commercial profit. Its import price began at 9s. 6d., or more, per pound in 1922, then 8s. 4d. in 1923, coming down gradually under the shadow of the approaching British product (for these things get known), so that by the time Scottish Dyes put this colour on the market in August 1928, the price had suddenly dropped from 4s. 7d. to 3s. 11d., indeed the market had been so rushed just at that time by the drop in price, that the imports rose to nearly double in the immediate period and we were left with almost no demand for a considerable time. We put it on the market at the same

price as the latest German quotation of 3s. 11d., and our price for bulk quantities is now in the neighbourhood of 3s., and we get the total home trade.

But I want to show what this intervening period of unreadiness has cost us British users in this one colour alone, and you may take it as an index of what it is still costing us in these colours we are not yet making; and, what is vastly more important, as an index of the tremendous additional sum our total colour demands would have cost this country under post-war conditions had we not been in a position to supply 90 per cent. of the home demands.

In the figures I give, I am going to include with Brown R and G, Golden Orange 3 G, as this is a closely allied colour of practically the same chemical composition and cost.

#### **What We Pay for Imports**

Since 1922 we have imported of this colour (in the three varieties) a total of 818,283 lb. These have cost us at the prices charged from time to time (including the large quantities allowed in at the latest and much reduced price) the gross total of £208,658. Now, as I have said, with the conditions under which the Germans had been manufacturing all these years, I know that a most liberal valuation, and one allowing them a very substantial profit indeed, would be a selling price of 3s. a lb. Thus, if we calculate 818,283 lb. at 3s. a lb., it gives a gross amount of £122,742. The amount we actually paid, as I have shown, was £208,658, being a sum of £85,916 in excess of what, I am sure, under their conditions of manufacture was already a high economic price. It would be safe to say that their trade with us in this one colour alone has realised for them considerably over £100,000 net profit.

I think these figures speak for themselves in terms too eloquent to need comment as to their significance. We must not complain. The colour was a splendid chemical achievement and their post-war charges indicated their very obvious policy. But that vast sum represents the special toll which as users we had to pay within a few years for practically one colour. For there is no doubt that had we had time or opportunity to solve that particular problem in 1922, instead of in 1928, that sum of £85,916, and more, would have been saved to British users.

That little story is, as I have said, a very interesting and illuminating epitome of the many phases of the dyes question as it affects this country to-day, and, in particular, is a sure index of the enormous value our home industry has been as a check on excessive charges from abroad over the whole range of dyestuffs; and it shows in a forceable way the necessity of makers here putting themselves in readiness to cover the whole field of the dye industry with the least possible delay.

#### **Has the Textile Trade Suffered?**

But, against this, I can hear some users argue that there was surely a time when they could have procured from abroad colours common to the manufacture of both countries at prices much lower than the home quotation, and that the textile trade in this way suffered seriously in consequence. In certain groups of colours I agree that, for a period, a condition of this kind existed. While we were quoted high and, as I have shown, quite uneconomic prices for colours that were not made here, users were offered prices for certain dyes made here which, at the time, would have been quite impossible for the home dyemaker to meet. That was a considered policy on the part of the Germans to upset the users here and to strike a blow at the operations of our Dyestuffs Act. It caused much unsettlement, and at the time made some bad feeling between users and makers here; so much so that the continuance of the Dyestuffs Act was for a time in jeopardy and was the subject of special discussions in Parliament. But the loyalty and goodwill of the majority of the users prevailed, and the establishing by the Licensing Committee of a price factor put an end to this overt attack, with the result that dyemakers here got faith to proceed and, with bulk production and experience, have now got to the stage of being on a fairly comparable basis in price with the foreign producers in the colours they make. During the past two years very few licences have had to be granted on the grounds of price, and the recent comments on this aspect of the trade by Sir Henry Sutcliffe Smith, chairman of the Colour Users' Association, are ample testimony to the fact that on the question of price, in the colours made here, the industry is on a fairly sound basis, and users are being supplied on terms comparable with their foreign competitors.

In retrospect of these last ten years, therefore, while there have been difficulties and some hardships to face on the part of the users, in giving to the makers in this country time for their processes to mature, I am sure the bulk of the users will agree that on balance it has been enormously to their gain to have the services of a home dye industry.

As the idea persists, however, among an aggressive minority that the industry has been fostered at great sacrifice by the users, and that it has been a distinct factor in the continued depression of the textile trade, I should like to suggest one or two broad tests on this very important point. If the lack of dyes or high prices have been a serious obstacle, this should be reflected in the returns of those branches of the trade dependent mainly on their dyes content, as distinct from other branches not dependent on dyes. We find the recent report of the Government Cotton Inquiry most valuable and illuminating in this respect. It analyses and tabulates the export returns of the various sections of the textile trade, and these are the figures. In Grey, unbleached cloth (using no dyes) the exports decreased from 2,357,492 thousand linear yards in 1913 to 954,823 in 1929. In Bleached cloth (also with no dyes) the decrease was from 2,045,252 thousand yards in 1913 to 1,288,273 in 1929. Printed goods (partially coloured only) decreased from 1,230,754 thousand yards in 1913 to 551,706 in 1929; while dyed and dyed-yarn goods (wholly coloured) decreased only from 1,441,754 thousand yards in 1913 to 970,049 in 1929. Indeed the sterling value of the dyed goods showed an actual increase in the period, the figures being £26,569,000 in 1913 against £33,914,000 in 1929.

These most striking figures offer much food for reflection to textile people, but surely the last inference that could be drawn from them is that the dyes supply has been a deterring factor.

### **1931 Spring Colours**

#### **Shades Chosen by American Associations**

EIGHTY new colours appear on the confidential advance Floss Card of the Textile Colour Card Association of the United States, it is announced by Margaret Hayden Rorke, managing director. For the trade promotion of colour correlation, and further to assist the textile and allied industries, the Spring Floss Card is released four weeks earlier this year. Indicated on the card are eleven shades suggested as ground colours for prints. The fashionable colours for daytime wear are grouped in ranges of three and four tones, while the outstanding feature of the card is a group of twenty-two nuances presented under the title of "Les Belles Couleurs Anciennes."

The Associated Dress Industries of America and the Allied Millinery Associations have chosen the colours that they will promote for the 1931 spring seasons from the advance floss card. The thirty-four colours selected by the Associated Dress Industries are as follows: *Blues*—Hilite Blue, Clematis Blue, Amulet Blue. *Greens*—Pistachio, Chukker Green. *Beige Browns*—Rose Cedar, Bisque Brown, Sea Sand, Noisette. *Rose*—Fraise, Framboise. *Grey*—Sky Grey. Also the entire group of twenty-two shades featured in "Les Belles Couleurs Anciennes."

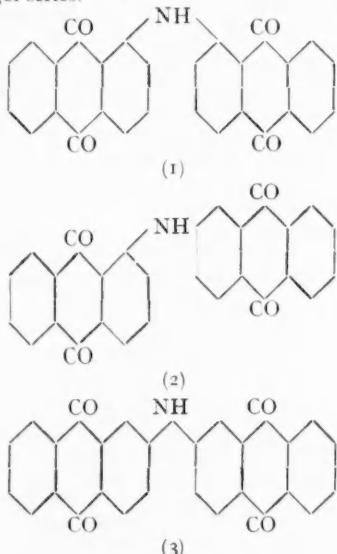
The thirty-nine official colours chosen by the Colour Conference Committee of the Allied Millinery Associations are: *Blues*—Hilite Blue, Clematis Blue, Amulet Blue, Guardsman Blue, Admiralty, Basque Blue. *Greens*—Pistachio, Maris Green, Chukker Green. *Beige-Browns*—Rose Cedar, Bisque Brown, Sea Sand, Mauve Bisque, Noisette. *Grey*—Sky Grey. *Red*—Talisman Red. *Rose*—Fraise. Also for sports wear, the twenty-two shades in the group of "Les Belles Couleurs Anciennes."

Nine colours for women's shoes are included in the 1931 Spring Shoe and Leather Card of the Textile Colour Card Association. These shades were chosen by the official colour committee of the Tanners' Council of America, the National Boot and Shoe Manufacturers' Association and the National Shoe Retailers' Association. The new colours are: Sea Sand, a new light beige with a sandy cast; Putty Beige, a neutral medium beige, also sandy in cast, and lacking the yellowish note of previous seasons; Indies Brown, a neutral brown; Swagger Brown, a medium russet brown; Sky Grey, a true medium grey, a new colour in shoe fashions; Paddock Green, a dark green, slightly yellower in cast than the greens of previous seasons; Grenat, a deep rich red.

## Basic Intermediates for Dyestuffs : No. XXXVIII.—Anthraquinone Halogen Derivatives, and the Dianthraquinonyl-amines—Part 1

By "Consultant"

THE importance which vat dyes have assumed in the preparation of coloured fabrics is increasing rapidly, on account of the fastness to light and laundering which they exhibit, and the intermediates for the preparation of the dyes of the anthraquinone class, from which many of these vat-dyes are derived, have become of considerable interest. The halogen derivatives of anthraquinone are valuable for the preparation of this class of dyestuff on account of the labile nature of the chlorine atom, which will react with the amino group giving rise to the anthraquinonylamine derivatives, many of which are important dyestuffs. There are three possible dianthraquinonylamines (dianthrimides, as they are sometimes termed) : the 1 : 1<sup>2</sup>, 1 : 2<sup>1</sup> and 2 : 2<sup>1</sup>, the formulae of which are shown below. Of these three, the 1 : 2<sup>1</sup> derivative is the more important, as derivatives of this compound constitute the valuable Algol series.



So many types and series of derivatives are available in this group of compounds that it would be impossible to describe them all in detail, so that for the purpose of this series of articles the preparation of Algol Red B, through the medium of 1 : 2<sup>1</sup>-dianthraquinonylamine (2) will be described.

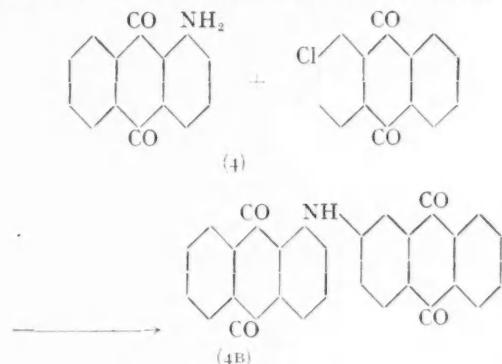
### 2-Chloranthraquinone

The raw material for the production of 2-chloranthraquinone (3) is sodium of anthraquinone-2-sulphonate, the production of which was described in No. VI of the Basic Intermediate Series. This substance ( $\frac{1}{2}$  cwt.) is dissolved in water (168 gall.) with the aid of hydrochloric acid (density 1.16; 17 gall.) and the temperature of the solution is then raised to 100° C. The conversion of this sulphonate salt to the chloro derivative depends on the fact that nascent chlorine will replace the  $-\text{SO}_3\text{H}$  group by chlorine, liberating sulphuric acid and hydrogen chloride at the same time. To bring about this reaction sodium chloride ( $\frac{1}{2}$  cwt.) is dissolved in hot water (56 gall.) and the solution slowly run into the warm acid sulphonate solution. The end of the reaction can be seen when no more chloranthraquinone separates out. The latter compound is separated off and washed; it needs no further treatment.

### 1 : 2<sup>1</sup> Dianthraquinonylamine

Several methods have been proposed for the manufacture of this compound. The first method, that of the inventor, was the condensation of 1-chloranthraquinone with 2-aminoanthraquinone in the presence of sodium acetate, cuprous chloride and some inert anhydrous solvent. The yield was,

however, poor, and later it was shown that if the positions of the chlorine atom and amino group were reversed, then the reaction proceeded much more easily. Accordingly, 2-chloroanthraquinone (3) and 1-aminoanthraquinone (4) are heated with



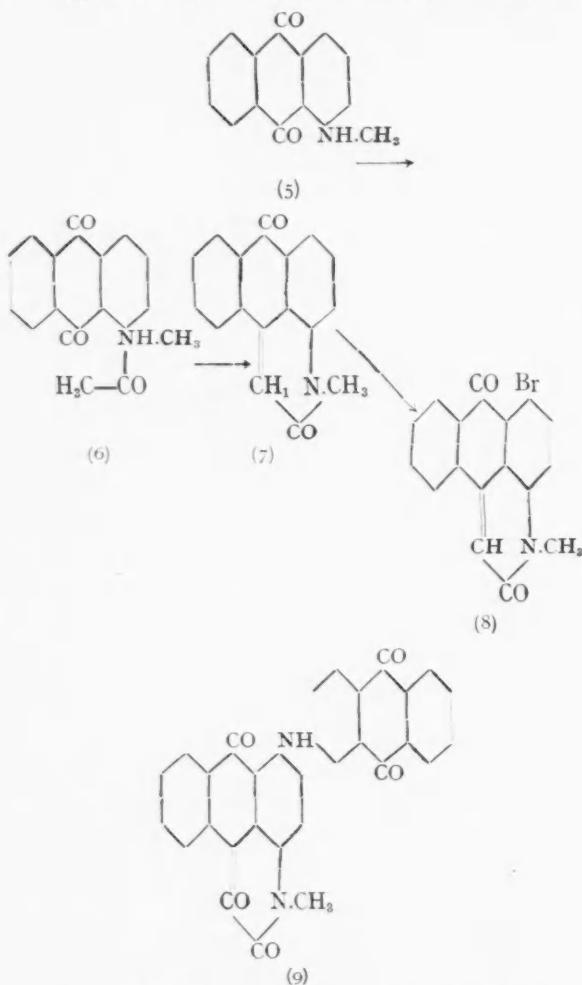
a mixture of finely ground sodium acetate and cuprous chloride in nitrobenzene suspension. The equimolecular proportions of the chloro compound and amine are used, together with one-twentieth of their weight of the cuprous chloride-sodium acetate mixture. The method just described has been to a large extent superseded by a process in which the conversion of the anthraquinone sulphonic acid to the chloro derivative is dispensed with. In this process (D.R.P. 201,327 and 216,083) the sodium salt of anthraquinone-1-sulphonic acid is mixed with an equal weight of 2-aminoanthraquinone and the mixture autoclaved with half its weight of potassium carbonate (anhydrous) and ten times the weight of nitrobenzene. The autoclaving is carried on for about four hours at 280° C. and the separation of the diamine compound may be effected by blowing off part of the nitrobenzene into a condenser as the autoclave is "let down," and removing the remaining nitrobenzene with steam. The residue is washed and dried. It has a small use as Algol Orange R. (4B).

### Algol Red B

For the preparation of Algol Red B the raw material is methylaminoanthraquinone (5) which is now prepared in one stage from the corresponding sulphonic acid. The addition of an oxidising agent to the mixture is necessary in order to account for the sulphurous acid produced in the process, which if not removed reduces the yield. In the Badisch process the anthraquinone-1-sulphonic acid (sodium salt) is heated with seven times its weight of 5 per cent. methylamine solution in water, with one-seventh of its weight of potassium bromate which acts as the oxidiser. The heating is best done in an enamelled autoclave with a good stirring, and the reaction takes between five and seven hours at a temperature of 160° C. The methylaminoanthraquinone separates out as a sandy crystalline powder and needs only to be filtered off and washed with water for use in the next stage of the synthesis.

The next step in the preparation of Algol Red B, the acetylation of aminomethylanthraquinone, is best done in oleum solution. The aminomethylanthraquinone is dissolved by stirring in the cold with ten times its weight of 22 per cent. oleum. Acetic anhydride, equal in weight to the amino compound used, is then added and stirring continued until a sample shows that acetylation is complete; the stirring is best done at 40° C., a process which shortens the time required for the completion of the reaction. The compound may be isolated by pouring on to ground ice, avoiding any rise in temperature, which causes hydrolysis. In the case of the production of Algol Red B, there is no need to isolate the acetyl derivative, since on warming the acid solution, dehydration takes place with the formation of N-methylanthrapyridone (7), which can then be isolated by pouring into water. There are alternative

methods of the preparation of N-methylanthrapyridone, such as the isolation of the acetyl derivative and heating with zinc chloride, as has already been described, and these, although more prolonged, give a better yield. The remainder of the process consists in the bromination of the N-methylanthrapyridone to give the 4-bromo-N-methylanthrapyridone



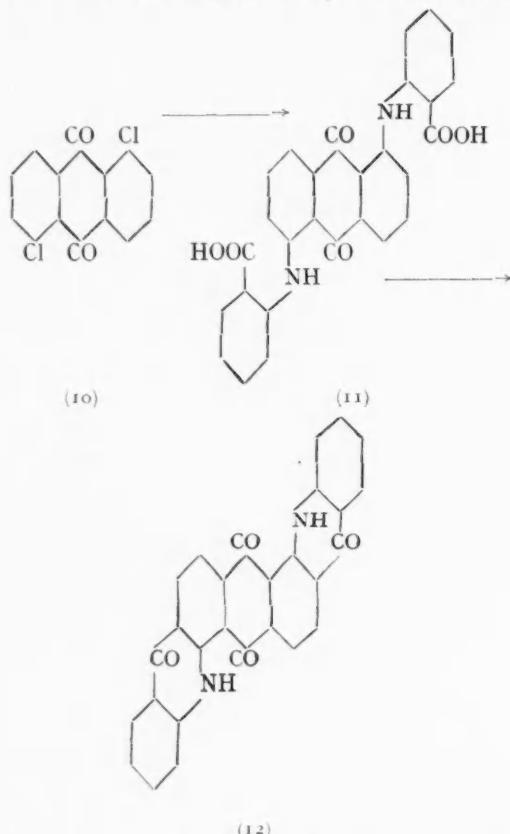
(8), and the condensation of this with 2-aminoanthraquinone in precisely the same manner as that described above for dianthraquinonylamine. The product, Algol Red B, is shown in (9).

#### Dichloranthraquinone and the Indanthrenes

Among the more interesting of the dichloranthraquinones the 1:5-compound has been selected for discussion. It may be readily prepared on a large scale by a process analogous to that already described for the preparation of the chloranthraquinone. In this case, however, the sodium salt of the 1:5-anthraquinone disulphonic acid is dissolved in boiling dilute hydrochloric acid. Hydrochloric acid of density 1.16 (11 gall.) and 110 gallons of water will be required to dissolve  $\frac{1}{2}$  cwt. of the sodium salt. The replacement of the sulphonic acid groups by chlorine is effected by the addition of sodium chlorate (1 cwt.) dissolved in water (80 gall.). This addition is made slowly, and the dichloro body separates out as formed, the boiling and addition of sodium chlorate solution being continued until no further dichloro compound separates. It is then filtered off and dried.

As a typical example of the formation of other intermediates from 1:5-dichloranthraquinone the preparation of Indanthrene Violet RN has been selected. This dye, although not perhaps of overwhelming commercial importance, excellently illustrates the formation of fused rings of this series. The first

process is the condensation of the dichloroanthraquinone (10) with anthranilic acid to give anthraquinone-bis-anthranilic



acid (11). This process is performed by boiling the dichloro acid with eight times its weight of nitrobenzene, together with potassium anthranilate (one-sixth the weight of the nitrobenzene). In addition, about one pound of precipitated copper oxide is added for every fifteen pounds of dichloro acid in the charge. The whole is boiled until the colour deepens no further. The bisanthranilic acid derivative of anthraquinone separates as a dark violet powder, which is filtered off, blown with steam to remove nitrobenzene, and washed successively with dilute nitric acid and water. To convert this to Indanthrene Violet RN the dry powder is dissolved in 100 per cent. sulphuric acid (ten parts) and heated at 100-110° C. until the colour has changed to an orange red; when this stage is reached the Indanthrene Violet (12) is isolated by pouring into water and filtering in the usual way.

#### New Dye Tub Control

THE Foxboro Co., Mass., U.S.A., has recently completed a new dye tub control system which fits the modern trend in piece dyeing. This controller takes the preheated dye water being delivered to the machines, raises it to the exact temperature needed, and holds it at this temperature for a definite time predetermined by the dyer. When the time is up the instrument shuts off the steam and turns on a signal light.

In addition to the temperature control unit, the instrument is equipped with a time scale and a movable index, which is set by the operator to the point representing the total time for the dye operation. If more time is required, the index is set for the increase in time, and the steam is automatically turned on, the light is extinguished, and the process continues. The operator always has before him as a guide a complete record of each operation in time and temperature, giving unlimited possibilities for matching shades. The unit is furnished complete, mounted on an ebony asbestos panel with lamp bracket and accessories.

## Dyes and Their Application: Recent Technical Progress

By L. J. Hooley

RECENT advances in the production of new synthetic products, such as cellulose esters and ethers, synthetic resins, and new condensation products having dispersing and similar properties, have provided starting points for many new processes in dyeing, printing and finishing. Dispersing and wetting agents still occupy a prominent position, and although the new ones follow the general lines of the old they are not without interest. Some of the more recent ones are given below.

### New Synthetic Wetting Agents

(a) n-Butyl alcohol is heated with naphthalene- $\beta$ -sulphonic acid in sulphuric acid monohydrate at about 60-70° C. (I.G.F.A.-G., G.P. 459,605). This is apparently a selection from the many alkylnaphthalene sulphonic acids which have been covered by the I.G.F.A.-G.).

(b) Aromatic compounds are treated with aromatic sulphonic esters of aliphatic alcohols, for the introduction of alkyl or aralkyl groups, either in the side chain or nucleus (Foldi, B.P. 319,273).

(c) Furfuraldehyde is condensed with naphthalene or other ring compounds and sulphonated (Brit. Celanese, G. H. Ellis, H. C. Olpin and E. W. Kirk).

(d) Benzoin, an aromatic sulphonic or carboxylic acid, and an alcohol are condensed (S.C.I.B., B.P. 319,249).

(e) Quaternary ammonium bases having at least one aromatic substituent and cyclo-aliphatic or hydroaromatic acids are condensed (I.G.F.A.-G., B.P. 316,090).

(f) Paraffin wax is oxidised and then emulsified with a soluble salt (I.G.F.A.-G., B.P., 322,429).

(g) A sulphonated mixture of aromatic hydrocarbons and a natural resin is condensed with an aralkyl halide (I.G.F.A.-G., B.P., 320,190).

Esters of adipic acid or substituted adipic acids have been found to have wetting properties (Böhme A.-G., B.P. 307,397). The ethanolamines and glycol ethers have been mentioned in previous issues of the DYESTUFFS SUPPLEMENT.

As regards the use of the wetting agents, aromatic sulphonic acids mixed with sulphonic acids from fatty acids with more than eight carbon atoms are specially resistant to saponification and also soften textiles (H. T. Böhme A.-G., B.P. 298,560). Halogen substituted sulpho-acids of aliphatic or hydroaromatic substances containing at least 10 carbon atoms are recommended by the Oranienerger Chem. F.A.-G. (B.P. 289,863) and the sulphuric esters of amides or anilides of fatty acids of high molecular weight by the H. T. Böhme A.-G. (B.P. 318,542). Substitutes for Turkey red oil, more suitable for use, because more stable, in acid and alkali baths, are obtained by sulphonating a fatty acid at low temperature with excess of sulphuric acid (H. T. Böhme A.-G.).

The commercial names, chemical composition, relative effectiveness and methods of testing wetting agents are outlined by A. Landolt (the *Melliand* 1, 243-8). A summary of the patent literature is given by J. P. Sisley in the *Revue Générale des Matières Colorantes*, and a discussion of various new detergents, thickeners and wetting agents by A. Noll in *Seide*, 34, 242-7.

### Dyeing

For dyeing with vat dyes, where there is difficulty with penetration, as in tightly twisted fabrics or dressed fabrics, a substance with a bleeding or running, as distinct from a wetting, influence, such as a soluble carboxymethyl cellulose or sulphonated oleic acid, is used, being padded on with the dyestuff and the goods then run through a reducing bath (I.G.F.A.-G., B.P. 305,230).

Instead of the usual alkali, an amine or heterocyclic base can be used in the dyeing of vat or sulphur colours (I.G.F.A.-G., B.P. 320,978).

### Printing

A method of printing analogous to that padding method in which the unreduce vat dye is used, is described by M. Jeanmaire and L. Ebersol, the thickened dye being printed on the wet fabric and then jigged in an alkaline reducing vat (*Chimie et Industrie*, Spcl. No. Feb. 1929, 493-4). G.P. 486,596 claims the use of iron-tetracarbonyl, potassium carbonate and glycerol and starch as a reducing agent in vat

printing. The resulting iron oxide is removed in a bath of oxalic acid (I.G.F.A.-G.).

Fabrics with printed patterns on each side, which are in register, but in different colours on the two sides, are prepared by printing one side with a coupling component, drying and printing the other with a different coupling component, again drying and then passing the cloth through a diazo solution (C. P. A. and J. D. Webster, B.P. 326,823).

### Discharge Printing

Titanium salts have comparatively recently been mentioned for reducing vat dyes, a salt of trivalent titanium in an alkaline medium is more recommended for reducing on the fibre. A protective colloid can also be added together with sodium formate or lactate (I.G.F.A.-G., B.P. 330,646).

### Reserves

Sodium meta-nitrobenzenesulphonate, which has already been used to prevent marking off in the kier boiling of vat dyes, its action being presumably to counteract reduction, has now been utilised in a new way in printing woven goods. The fabric is impregnated with the sulphonate from a hot aqueous vat, with size if desired, the woven material is then printed with a reduced vat dye paste, dried, steamed and oxidised.

Certain dispersing agents will act as reserving agents for animal fibres, thus the product from condensing a natural resin with an aralkyl halide and sulphonating can be used in reserving wool when using direct dyes with cotton (I.G.F.A.-G., B.P. 304,742) a sulphonated phenol or naphthol condensed with a halogenated aralkyl halide behaves similarly and in addition can be used in tanning. Phenol sulphonic acid condensed with o-chlorbenzoyl chloride is included in the example (I.G.F.A.-G., B.P. 320,056).

For reserving silk in the presence of wool an organic sulphonic acid which will precipitate glue or gelatine from an acid solution is described in B.P. 297,124 (I.G.F.A.-G.).

A simple steaming apparatus for small scale operators and suitable for use in the East, consists of an upper container in which the fabric printed with a vat dye is surrounded with sand or similar material to keep out air and a lower one in which the steam is generated (I.G.F.A.-G., B.P. 11,476/29). Candite V, a mixture of hydrosulphite and glucose is recommended for decreasing the attack of copper rollers in printing.

Mono- or di-methylol urea can be used in printing to give clean prints or in the absence of colour to give lustre and damask effects (B.P. 328,978, I.G.F.A.-G.).

J. G. Grundy classifies and tabulates the main groups of dyes which are suitable for the production of effect threads in *J. Soc. Dyers and Col. 1929, 45, 142-8*.

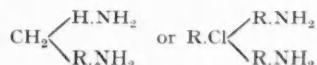
### Body Colours

Some interesting miscellaneous applications of colours as pigments may be mentioned. In B.P. 324,864 (G. Fugmann), a stone such as marble is coloured either on the surface or throughout by using metal compounds of dyestuffs dissolved in organic solvents with the addition of esters of organic acids. For example, a mixture of copper nitrate, amyl acetate and cellulose ester may be used. In F.P. 570,093 (Soc. Alther and Guex and W. Hugentobler), cadmium yellow is printed on to fabrics in a paste with linseed oil diluted with turpentine and glycerine.

A metal salt of a triphenylmethane dyestuff having a sulphonic acid group ortho to the central carbon atom is suitable for fast-to-vulcanisation colours on rubber. Alternatively, a metal salt of an aminoanthraquinone carboxylic or sulphonic acid may be used (B.P. 277,034, I.G.F.A.-G.).

### Oil Soluble Colours

Tetrazo compounds of condensed amines of the general formulae



where R is an unsulphonated aryl group coupled with a non-sulphonated azo compound, are soluble in oils such as linseed oil giving solutions suitable for colouring paper (B.P. 301,726, Geigy).

## Dyestuffs Markets: The Month's Business in Review

From Our Own Correspondents

### Lancashire

WHILST Lancashire is still struggling along in the rough seas of the textile depression there is another question to which more and more thought is being given—whether the Dyestuffs Act is to be allowed to expire next year or to be renewed for a further period. There are strong arguments to be brought forward on either side. Some, at any rate, of the dyers maintain that the foreign dyestuffs firms should be granted free and unimpeded access to English markets on the ground that by the present Act they are debarred from purchasing the latest novelties and improvements in the way of dyestuffs that have been invented abroad. Perhaps before the Dyestuffs Licensing Committee had got properly into their stride, unfortunate delays may have taken place, but this state of affairs has long been passed. In spite of the rapid growth of the production of British-made dyestuffs, both in quantity and in quality, licences were obtained during last year for no less than 5½ million lbs. weight of dyestuffs and intermediates, of a value of over £1,000,000.

### What Dye-makers Have Had to Face

When the Dyestuffs Act was first passed, it was to run for a period of ten years. Ten years of protection to allow the British dyestuffs industry to perfect the manufacture of what are perhaps the most complicated bodies known to organic chemists. The protection given to a patentee under ordinary circumstances runs for 14 or 16 years, and may under certain adverse conditions be extended for a further period of years. The advocates of the continuance of the Dyestuffs Acts claim that they have had to face circumstances much more adverse than those for which a patentee would most certainly be granted an extension. During the first two years of the Act, foreign dyestuffs of over £7,000,000 in value were imported, partly owing to the freeing of the ports, and partly in the form of reparation dyes. These dyes were not used up until well into 1923. At this time the general trade depression began, and has continued ever since, especially in the textile industries, who are the greatest dyestuff consumers. Consequently, the dye-makers have not been able to enjoy the benefits of protection to anything like the extent anticipated. Millions of pounds have been expended in plant and building by the dyestuff manufacturers, and large numbers of chemists and engineers have been trained up especially for the purpose of carrying on a permanent dyestuff industry in this country. The immediate effect of opening our ports to foreign dyestuffs would be the scrapping undoubtedly of the greater portion of all these valuable works and their specially trained staffs, for there is no doubt that the foreign dyestuff makers would at once open a campaign to capture the market for all the latest and improved types of dyestuffs. These would be offered at a price with which the works here could not compete. With the British works out of action, monopoly would pass once more to the foreigner and prices would rise once more in accordance with the new balance of power. The large temporary gains made by the users here would indubitably be wiped out, in order that the foreign dyestuff makers could recoup themselves for the expenses of the price cutting campaign.

### Future Progress

The suggestion made by Sir Henry Sutcliffe Smith to impose a safeguarding tariff on certain agreed colours already being made here, would merely have the effect of denying all chance of future progress to our own chemists. They would almost certainly fall into stagnation and decay. Dr. James Morton, in his weighty address to the British Association—weighty from his wide experience, both as a user of dyes and as a manufacturer—claims that the Act should not only be renewed, but should be still further strengthened by the addition of a clause prohibiting the importation of colours from abroad that are merely the practical equivalents of colours already being made here—equivalents that are marketed with the sole object of avoiding exclusion under the present Act. Whilst on the part of a large number of dye users there is this strong feeling that the Act should be allowed to drop, and the importation of foreign dyestuffs be freed from all restrictions, those

who feel favourably disposed towards the continuance of some method of control consider that they, the consumers of dyestuffs, should not be forced to bear the whole cost of the measure, but that restriction of imports should be supplemented by a form of subsidy from the Exchequer, thus helping to spread the burden of maintaining the dyestuff-making industry in its present vigour over the whole nation, instead of confining the cost to those least able, under present circumstances, to carry the entire load.

### Scotland

THE news this month has been a mixture of little of the cheerful with a good deal of the depressing. On September 20 the large silk dyeing works which have been built at Balloch by Loch Lomond were opened by Sir John Gilmour, Bt. The works are very finely built for the purpose, occupy about two and a half acres and are estimated to have cost nearly a quarter of a million. Against this there have to be set down the winding-up of the Scottish Amalgamated Silks, Ltd., with its subsidiary company, Scottish Artificial Silks, Ltd., and the closing down of the Thornliebank Print Works. This last has been a great disappointment to Scotland, and the only bright spot in the outlook is the information that the works will be reopened if trade improves sufficiently.

In the south the tweed trade is much the same as during the last few months. The generally unsettled conditions at present are having some adverse effects on repeat orders for the winter season 1930-31, although the spring orders for 1931 are a little more satisfactory, especially in the higher classes. Sales of dyestuffs show little change.

### British Colour Council

A COLOUR Selection Committee was appointed at the first meeting of the Board of Management of the British Colour Council held in London on Tuesday. The immediate object of this Committee is not actually to select colours, but to settle the principles upon which colour selection will be determined.

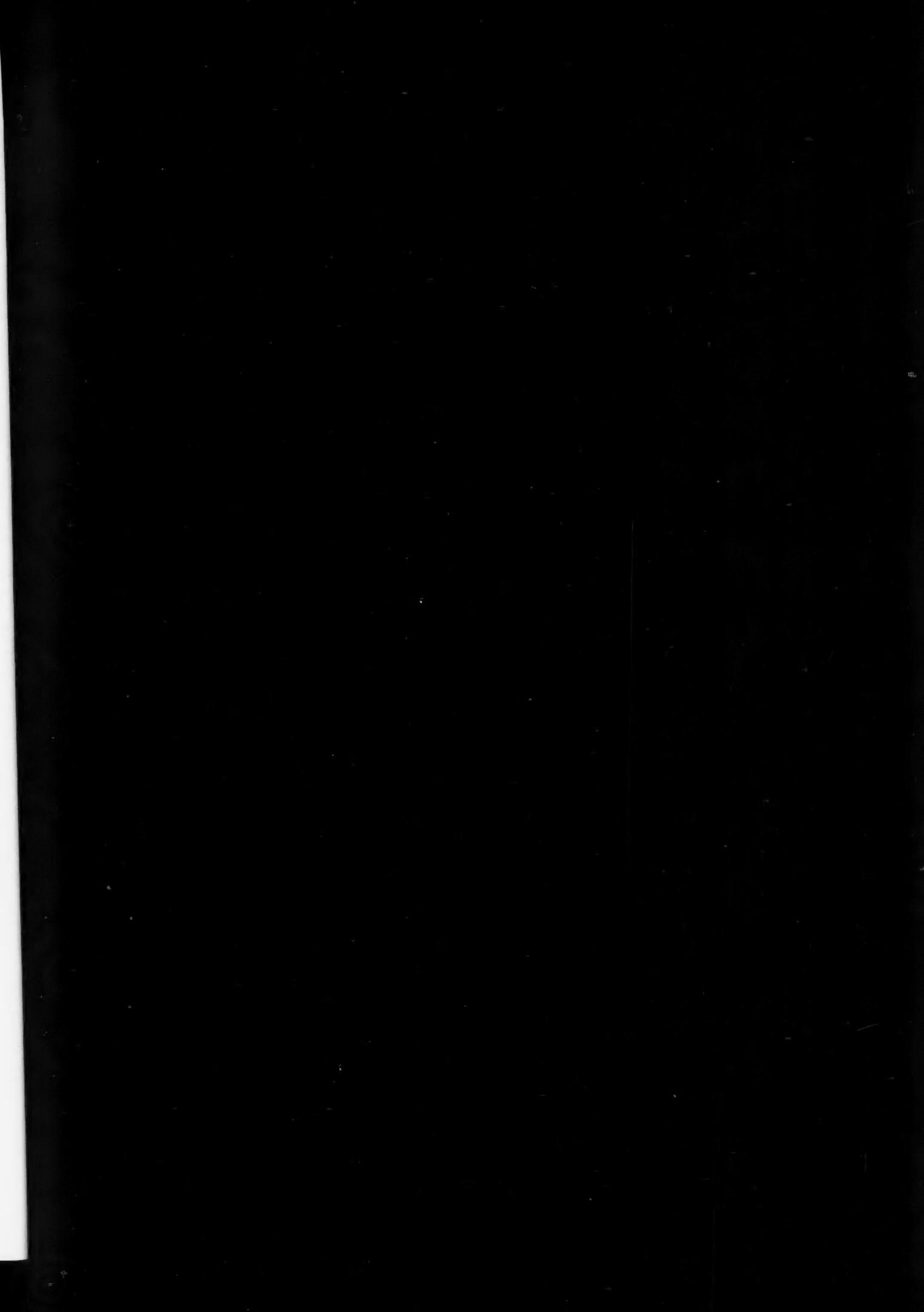
The Colour Selection Committee, which has power to co-opt members, was appointed as follows:—Mr. John Sharp (Bradford Dyers' Association), chairman; Mr. C. B. Gwynne (B.D.A.), Mr. Holbrook Jackson (National Trade Press, Ltd.), deputy chairman; Mr. C. M. Whittaker (Courtaulds, Ltd.), Mr. John Kerr McCallum (J. and J. McCallum, Paisley), Mr. J. H. Mortimer (J. H. Mortimer, Ltd., Bingley), Mr. Edward H. Symonds (Revile, Ltd., London), deputy chairman; Mr. Cecil J. T. Cronshaw (Imperial Chemical Industries—Dyestuffs Group), Mr. A. W. Tudor (Drapers' Chamber of Trade).

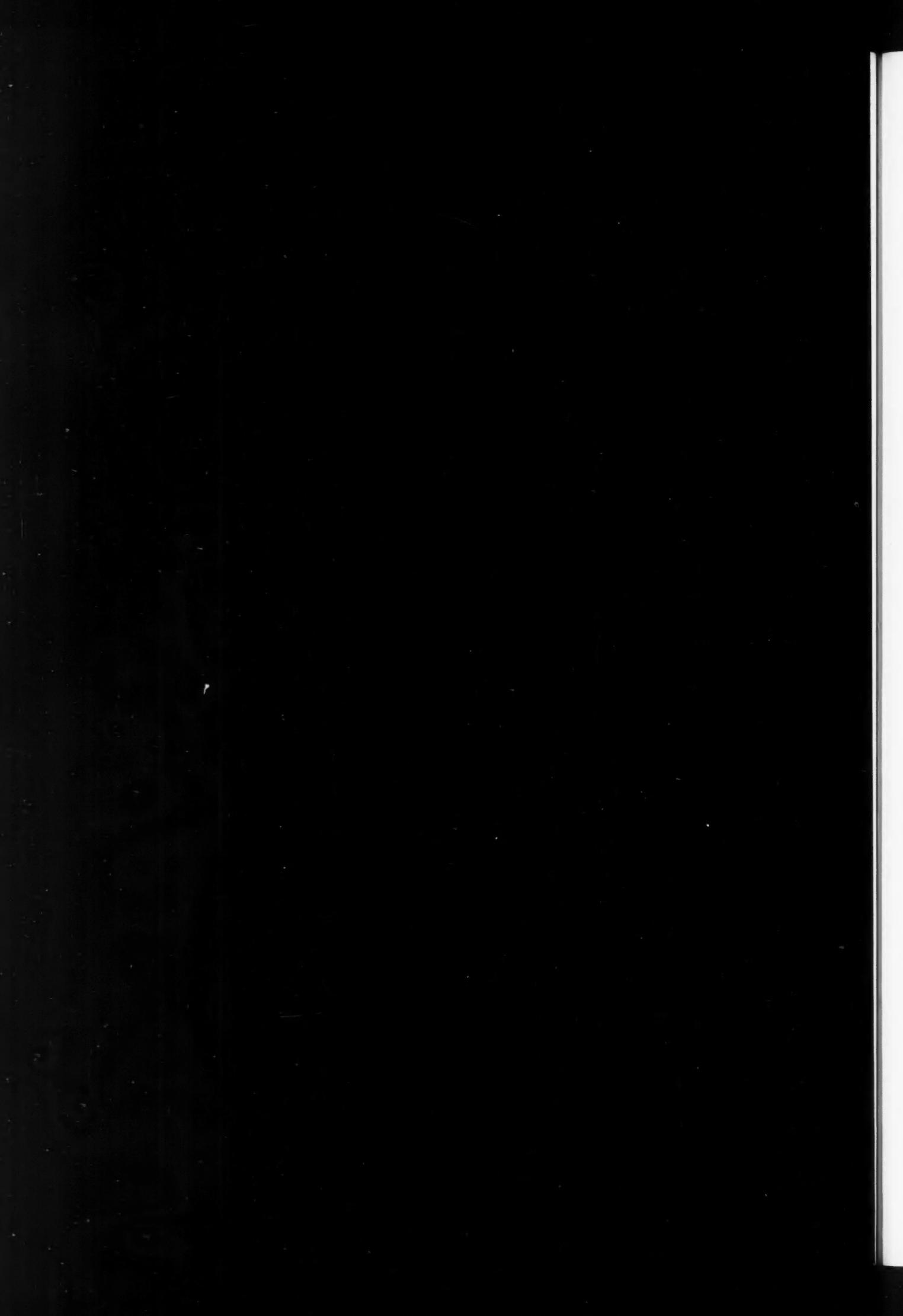
Another Committee, appointed to deal with membership and to make arrangements for premises and staff, consists of Mr. Sharp, Mr. Holbrook Jackson, Mr. Symonds, and Mr. Herbert Kay, hon. secretary of the Council.

### Russian Dyestuffs Production

ACCORDING to official sources, the total production of dyestuffs in Soviet Russia during the fiscal year (October 1 to September 30), 1927-28, totalled 10,793 metric tons and during the first half of the ensuing fiscal year, 1928-29, 6,423 metric tons. Production statistics of the more important classes of dyestuffs were:—

|                                 | First Half<br>1927-28 | 1928-29     |
|---------------------------------|-----------------------|-------------|
|                                 | Metric tons           | Metric tons |
| Sulphur black dyes, in paste .. | 7,901                 | 4,726       |
| All other sulphur dyes .....    | 363                   | 210         |
| Alizarin .....                  | —                     | 26          |
| Acid dyes .....                 | 536                   | 295         |
| Basic dyes .....                | 73                    | 37          |
| Impregnating dyes .....         | 140                   | 76          |
| Fur dyes .....                  | 30                    | 27          |
| Nigrosine .....                 | 251                   | 148         |
| Dye substances .....            | 1,499                 | 878         |





# Dyestuffs Monthly Supplement

Published in the second issue of "The Chemical Age" each month

Communications relating to editorial matter for the Dyestuffs Monthly Supplement should be addressed to the Editor, THE CHEMICAL AGE, Bowyer House, 154, Fleet Street, London, E.C.4. Advertisement matter, subscriptions, etc., should be sent to the Manager. The Supplement is devoted to the interests of both manufacturers and users of dyestuffs, and contributions will be welcomed.

### New I.C.I. Colours

Imperial Chemical Industries announce the production of five new colours—Coomassie Brilliant Blue FFS and Altex Union Orange G, Bordeaux B, Nigger Brown G, and Helio R.

Coomassie Brilliant Blue FFS, of which some delightfully bright examples appear on the pattern card, is an addition to the existing range of acid colours. It gives bright pure blue shades of excellent fastness to washing and very good fastness to milling, perspiration, carbonising and stoving. The colour is eminently suitable as a component of bright blue or bluish violet shades particularly on rags, loose wool, and slubbing. It may be applied in combination with chrome dyestuffs and in circulating machines. It possesses excellent affinity for wool or silk from a neutral dyebath which, combined with the valuable property of non-staining cotton or artificial silk, makes it of special interest for garment dyeing or union dyeing in general. It is described as very suitable for dyeing either tin weighted or unweighted silk, giving bright blue shades of very good fastness to water and washing.

Of the Altex Union Colours, of which four new varieties are now available, it may be said that they are capable of dyeing mixtures of all the various textile fibres—cotton, wool, silk, viscose, and acetate silk—and the original members of the series have found great favour amongst the garment dyeing trade. These dyestuffs are prepared for the dyeing of mixed fabrics, and should not be used for garments consisting entirely of acetate silk or other single fibres.

Altex Union Colours are dissolved by pouring boiling water over them, stirring at the same time. The solution obtained is added to the dyebath through a fine sieve. Dyeing is conducted at a temperature of 180-185° F., with the addition of 10-40 per cent. Glauber's salt or common salt dependent upon the depth of shade; dyeing is continued for 1 to 3 hours. The garment is then well washed and dried. No aftertreatment or development is necessary.

### London Lectures on Dyes

The London Section of the Society of Dyers and Colourists has arranged an interesting series of lectures for the coming session. On November 21 Professor F. M. Rowe will deal with "Properties of insoluble azo colours on the fibre"; on December 19 Dr. Callan, "Emulsifying agents, textile assistants and finishing materials: their examination and valuation"; on January 16, 1931, Mr. J. T. Holden, B.Sc., "Researches upon the laundering of fabrics"; on February 13, Mr. A. J. Hall, "Bleaching, dyeing and finishing processes, and their effect on finished goods"; and on March 20, Captain Whiteman, "Spray dyeing."

### Gloving Leathers

For many years alum-tanned or tawed leathers have been the main raw materials of the glove manufacturer. Such leathers possess the softness and stretch necessary for glove work. The dressing, or tawing, is, however, of such a nature that even a mere wetting-down with lukewarm water removes appreciable amounts of alum and salt, which results in a hardening of the skin. In order to dye such a leather satisfactorily, it is necessary to use natural dyewoods, which are rich in tanning, thus bringing about a tanning, as well as dyeing, action.

Largely on account of their washable character, chrome-tanned gloving leathers have recently come in favour, and with their advent new methods of dyeing have been found necessary. The tanning action of natural dyewoods (which in the case of alum leathers is of value) is, in the case of chrome-tanned leathers, a great drawback. The use of large amounts of dyewoods or dyewood extracts on chrome-tanned leather exerts a swelling action and also tends to give a tightened grain with much reduced stretching qualities.

### For Fuller Shades

For the fuller shades of colour, at present in vogue, the use of dyewoods alone on full chrome leathers would prove impracticable, and, in consequence, synthetic dyestuffs are being widely adopted. They are obtainable in every shade, and many possess good fastness to washing and light. The reds, greens and blues which have recently found favour can only be dyed with the aid of synthetic dyestuffs.

East Indian Sheep (Persians) are also now finding an extensive use in the gloving trades. They are usually stripped with alkali and well chromed before being dyed. For this class of work also a wide range of colours is available. Aldehyde leather is used extensively for white washable doeskin gloves. By the employment of such products as the Thionol colours it is possible to produce on aldehyde and chamois leathers shades possessing excellent fastness to washing without the objectionable dust which is so evident when colouring is done with clays and pigments.

The needs of the gloving trades have been given special attention by Imperial Chemical Industries, Ltd. This company is able to supply dyestuffs, fat liquors and chemicals suitable for all present-day requirements. Further, the technical staff and well-equipped laboratories at Blackley are always at the service of firms engaged in any branch of the leather trade.

### Dyestuff Licences for October

The following statement relating to applications for licences under the Dyestuffs (Import Regulation) Act, 1920, made during October, has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee:—

The total number of applications received during the month was 686, of which 585 were from merchants or importers. To these should be added six cases outstanding on September 30, 1930, making a total for the month of 692. These were dealt with as follows:—Granted, 650 (of which 634 were dealt with within seven days of receipt); referred to British makers of similar products, 32 (of which 21 were dealt with within seven days of receipt); standing on October 31, 10. Of the total of 692 applications received, 655, or 95 per cent., were dealt with within seven days of receipt.

### A New Library

Four new volumes of text-books on the printing and dyeing of artificial silks, issued by Imperial Chemical Industries, in an attractive style, appear to constitute the beginnings of a useful library of such pamphlets. They deal with "The dyeing of artificial silks," "Two-colour effects and solid shades on mixtures of acetate silk and viscose (or cotton)," "The printing of artificial silks," and "The discharge style on artificial silks." While small in bulk, these booklets include adequate practical treatment of

each subject, and will no doubt be welcomed by those engaged in printing and dyeing as convenient handbooks. We understand that free copies are available on application to the Publicity Department, Imperial Chemical Industries, Ltd., Imperial Chemical House, London, S.W.1.

#### Dyeing Acetate Silk

Taking the booklets in order, it may be said that cellulose acetate silk has by now established for itself a place as one of the most useful of textile fibres. In the early days of its manufacture, it resisted most obstinately all attempts at satisfactory dyeing, but the discovery of Duranol and Dispersol dyestuffs completely changed the situation. These dyestuffs not only dyed cellulose acetate satisfactorily, but made the production of fast colours on cellulose acetate easier to obtain than equally good results on any other fibre.

#### Two-Colour Effects

The Duranol and Dispersol colours have no affinity for cotton or viscose, but leave them unstained, and as a result it is possible to produce with them satisfactory two-colour effects on mixed fibres in one bath. This is made more simple by the fact that there are a number of Chlorazol colours which have been selected for their non-staining effect on viscose and cotton. Similarly, combinations of dyestuffs of these two groups can be used also for producing solid shades on mixed artificial silk fabrics. When it is desired to produce fast shades on fabrics of the type mentioned, Caledon vat colours can be used to dye the cotton or viscose portion, while Duranol colours are used for the cellulose acetate portion. These are the problems dealt with in the volumes on the dyeing of acetate silk and two-colour effects.

#### Printing of Artificial Silks

The use of artificial silks has grown so remarkably during the past few years that the printing of them has become a question of considerable importance. Viscose and similar regenerated cellulose types present comparatively little difficulty, since they can be treated in a similar way to cotton, which they resemble in affinity. Chlorazol, Basic and Vat colours can all be used with little difficulty. Cellulose acetate materials, however, were not quite so simple to handle when first introduced. The availability of the range of Duranol and Dispersol dyestuffs means that prints of excellent fastness to light and washing can now be obtained. Fast styles can also be printed with the aid of Caledon vat colours, while acid, basic and chlorazol dyestuffs can also be used in certain circumstances.

#### The Discharge Style

The fact that certain dyestuffs will colour one type of artificial silk and not others makes it possible to print (say) only the viscose portion of a mixed fabric containing two types of artificial silk. Interesting effects can also be produced by printing the viscose one colour and the cellulose acetate another. Textile printers use the discharge style to a very large extent, and in consequence this method must be given close consideration in connection with its application to artificial silks.

#### Selecting Suitable Dyestuffs

The principal classes of dyestuffs suitable for being discharged are Acid, Chlorazol, Azoic, Ionamine and Dispersol Fast dyestuffs, whilst the colours that withstand the process are certain Basic colours, the Duranol and the Caledon dyestuffs.

Actually it does not matter what the composition of the fabric is so long as the dyestuffs used for the preliminary dyeing possess the right characteristics. In spite of this generalisation, however, cellulose acetate materials provide some difficulty, owing to their resistance to wetting out, but a process has been patented by the British Dyestuffs

Corporation, Ltd., by the use of which the difficulty is overcome. As already stated, two of the booklets deal with the printing of artificial silks and the discharge style.

#### Dyestuff Fastness

It should be thoroughly understood (the *American Dyestuff Reporter* states) that there is no such thing as absolute fastness in dyes. If experience proves that a dye used for a certain purpose keeps its shade, it is said to be fast; at least, it is fast so far as that particular use is concerned. However, the same dye used for another purpose might prove to be extremely fugitive. At the present time, when we hear so much about fast dyestuffs, and the importance of them, we are apt to forget that the same questions have been in the minds of dyers ever since dyeing was first carried out. The ancient Egyptians were probably the first to start the demand for fast dyes. Moreover, they succeeded in obtaining a purple from a shellfish that has been able to withstand centuries. An examination of the old mummy cloths proves this. The dyeing of indigo was carried out in India hundreds of years ago. Indigo was, and still is, in some places dyed by the crudest fermentation vat methods. That crude dyestuff and the primitive methods of application marked the real beginning of the modern fast dyes. After the use of indigo was established, other natural dyestuffs, such as logwood, fustic and the mineral colours were discovered. In those days they were relatively fast; some of them were extremely fast to certain colour destroying agents.

#### Modern Dyes

Many of the dyes referred to would to-day be called very fugitive, simply because in relation to the newly discovered colours, they would not be nearly so fast. Then, too, there are now other considerations than sunlight to be taken into account when judging fastness. For instance, many of the modern dyestuffs, in order to be called fast, must be able to withstand the severe processes of crabbing, steaming, cross-dyeing, boiling with acids and kier boiling. The gradual introduction of these processes has been kept pace with by the gradual lengthening of the list of dyes—fast and other. Not only have new dyestuffs been discovered, but new classes of dyes, and new methods of application. With the greater use of the vat dyes, and especially in view of their coming use on wool, some may think that the ultimate in fastness has been obtained. However, past experience would seem to show that there can be no end to progress in this direction. Dyestuffs will continue to be improved just as long as they are used.

#### The Price Factor

Even though some laymen may ridicule the modern dyer's attempts at fastness, and point out the almost everlasting fastness properties of some vegetable dye, it is, of course, well known that modern science has produced an almost infinite number and variety of shades that are far faster than the natural dyes. It should be remembered that centuries ago price was a minor consideration. To-day we have to face the fact that increased production and low costs are paramount factors. Cheap goods are naturally to be coloured with cheap dyes, which will probably be far from the fastest obtainable. Modern fashions and customs, too, have taken a hand in the fastness requirements. Some garments go out of style after a short period of use. In such case, it would be useless to spend a large sum for colour when for a small sum colours could be used that would last for the length of time the fabric was in style. There is no doubt that fast colours can be obtained on any material if the consumer is willing to pay for them. He should realise, however, that when he buys cheap goods, manufactured on a production basis, they have been made with so small a margin of profit that expensive fast dyes cannot possibly be used.

## The Bleaching of Plain and Colour-Stripe Cotton Goods

By A. J. Hall, B.Sc., F.I.C., F.T.I.

Many interesting changes have occurred in the English bleaching trade from the time when James Watt first introduced from France the use of chlorine gas to the present time when bleaching powder and liquid chlorine are popular. Hitherto, bleaching processes have been carried on without more than a suspicion of scientific control. Recently, however, a number of researches have shown unsuspected factors which influence the rate of bleaching and its effect on cotton and dyes. Of particular importance is the pH of the bleaching liquor. This article deals with bleaching processes in the light of the new knowledge.

In spite of certain advantages, both actual and reputed, which are claimed for the use of other bleaching agents such as peroxides and ozone, only a very small proportion of cotton goods are bleached other than by means of chlorine compounds. Active chlorine in one or other of its various forms is cheap and readily available, and there thus appears to be no immediate likelihood of its displacement as the bleaching agent of first importance in the near future.

It is recorded (*A History of Bleaching*, S. Higgins) that the French chemist, Berthollet, first suggested the use of chlorine for bleaching purposes in about 1785. His own countrymen did not favour the use of chlorine gas for such a purpose, probably because of its dangerous nature, but through the introduction of James Watt (of steam engine fame) it was actually used some three years later in Glasgow for cotton fabrics and considered satisfactory. In these first bleaching operations it was customary to use as a bleaching agent either the gas itself or its solution in water. It is evident that in either case the operative was likely to inhale chlorine gas escaping from the bleaching chamber or liquor, and his trade was certainly a very unhealthy one. Subsequently it was discovered that the bleaching properties of chlorine were not lost if it was first dissolved in a dilute alkaline solution, although most of the dangerous escape of chlorine gas in bleaching was thus obviated, and the use of such a solution (that obtained with caustic potash was known and sold as Eau de Javelle) became general.

Afterwards, as is now well known, Tenant, of Glasgow, discovered that a satisfactory bleaching liquor could be made by absorbing chlorine gas in milk of lime. This was a distinct advance, but was soon followed by the manufacture of bleaching powder, for which dry lime was used as an absorbent. The use of bleaching powder for treating cotton goods has proved to be thoroughly satisfactory over many years, but to-day there is wide use of aqueous solutions of sodium hypochlorite (manufactured by electrolysis of brine), and also a reversion to the use of chlorine gas (in the liquid form which is thus under perfect control, and can be used in the bleach works for preparing sodium hypochlorite solutions).

### The Action of Chlorine

It is a curious fact that although active chlorine has been employed for bleaching cotton goods during about one hundred and fifty years, its action is not yet well understood. Chlorine affects both the cotton and the colouring matters in the cotton. The general aim of a practical bleacher is to destroy the colour and thus obtain a white without decreasing the strength or otherwise affecting the dyeing properties of the cotton. Experience has taught him how to do this with success, but of the chemical reactions involved he is usually mostly profoundly ignorant.

Within the past decade a considerable amount of research has been carried out in connection with the effect of the bleaching process on the cotton as distinct from its effect on the colouring matters. The results of these researches have without doubt led to a better understanding of the bleaching process, whilst at the same time they have indicated that a considerable amount of further research must be carried out. It is intended in this article to deal with the bleaching of cotton as revealed by the most recent investigations particularly as far as the bleaching process affects the cotton and dyed effect threads.

Excessive application of active chlorine to cotton results in certain changes which were clearly noted as early as 1883 by Witz. Briefly, these results include a loss of tensile strength, a decreased affinity for direct dyes, an increased affinity for basic dyes, the development of power to reduce various substances, but particularly Fehling's solution, an increased solubility in caustic alkalis with yellow colour, and a lowering of the viscosity of solutions of all its derivatives such as acetate

xanthogenate, and nitric ester. It is no wonder that with such a number of complex changes the investigation constitutes a wide field of inquiry.

Perhaps the most interesting fact which has emerged from recent researches on bleaching is that the acidity or alkalinity of the bleaching solution is of the utmost importance. In the bleach croft it has been generally known that if cotton goods proved difficult to bleach to a good white, the only way—though a dangerous one—was to "stink" them. In this process the bleaching liquor was slightly acidified. As a result, the odour of chlorine in the croft becomes very pronounced, and the cotton goods rapidly acquire that whiteness which is desired. The other disadvantage is that a loss of strength of the cotton may occur. But recent research (Clibbens and Ridge, 1927, 18, 141) has shown in a surprising manner that an exactly neutral solution of sodium hypochlorite may be much more effective than an acidified one—but it must be exactly neutral.

### Variations in Liquor

During the bleaching of cotton materials the liquor may become acidic, due to acidic substances being formed by oxidation of the impurities present, by absorption of carbon dioxide from the air, or by reason of acidic substances already present in the cotton. The pH of the bleaching liquor cannot therefore be assumed to remain the same as at the beginning of the bleaching process. Hence in an investigation of the effect of pH on the rate of bleaching a method must be adopted for

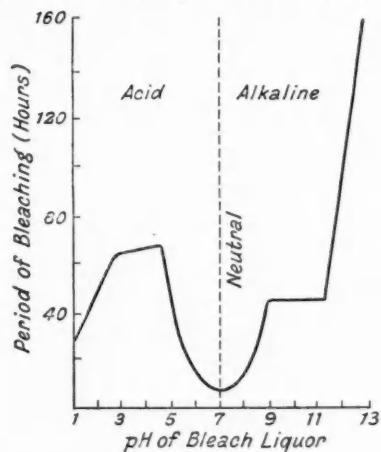


FIG. 1.

maintaining the pH constant. This may be effected conveniently by the use of suitable buffers.

In a series of experiments Clibbens and Ridge treated similar samples of cotton with bleaching liquors (prepared from sodium hypochlorite solution) maintained at various pH by means of buffering substances, and determined the time taken for destruction of one-half of the initial active chlorine; it may be assumed that the time taken under such conditions is proportional to the rate of bleaching. Their results are shown in Fig 1. The bleaching rate is obviously at a maximum when the pH = 7, which is the neutral point. If the bleaching liquor becomes very slightly alkaline or slightly acidic, the bleaching rate decreases rapidly. In certain circumstances the rate of bleaching may be decreased by ten times with such a slight change in the pH. Obviously in the large scale control of bleaching, careful attention must be given to the maintenance of the same pH throughout, otherwise, if this value changes one part of the fabric may be more thoroughly bleached than another.

Now it has been indicated above that the effect of a bleaching process on cotton may be measured indirectly by its effect on certain properties of the cotton. Among other properties it is here desired to draw attention to the effect of bleaching on the Copper Number of the cotton, on the affinity of the cotton for Methylene Blue, and also the viscosity of the cotton in cuprammonium solution. By determination of these effects it becomes possible to measure the rate of bleaching at different pH values.

The Copper Number of cotton is a measure of its reducing power, such power being increased by overbleaching and deterioration of the cotton. Actually the Copper Number is the number of grams of copper (cupric) which under certain empirically defined conditions is reduced to the cuprous state by one hundred grams of cotton. A satisfactorily bleached cotton should have a Copper Number not exceeding about 0.2; a very good bleached cotton may have as low a Copper Number

absorption of the cotton samples obtained as described above are shown in Fig. 2a. Once more the curve for pH=7 has the maximum steepness thus showing the high activity of the neutral bleaching liquor.

Finally the above results are confirmed by the curves in Fig. 2c which shows the viscosities of variously bleached cotton samples as described above. It is found that the more over-bleached is cotton the less is the viscosity of the solution it yields when dissolved in a cuprammonium solution. Furthermore, the viscosity is an excellent measure of the tensile strength of cotton—a low viscosity certainly indicates a low strength.

Thus by a number of methods it has been shown clearly that the pH of the bleaching liquor is very important, and in fact decides the efficiency and character of the bleach.

Those interested in large scale bleaching processes will probably be interested to know that it is not difficult to determine the pH of a bleach liquor. In carrying out such a determination it is necessary to destroy the oxidising character of the liquor without affecting its acidity or alkalinity, for the active chlorine especially under acid conditions would readily destroy any indicator added. This can be effectively done by adding a suitable quantity of a neutral solution of hydrogen peroxide when the following reaction occurs :



The pH may then be determined by addition of a suitable indicator (for example, the B.D.H. Universal Indicator), or the solution may be titrated in the usual manner.

In the bleaching of cotton piece goods it is sometimes found convenient to bleach after mercerisation. When this is done it must be remembered that the mercerised cotton is considerably more reactive towards a bleaching liquor than is non-mercerised cotton. The bleaching process must then be carried out in a shorter time or with a somewhat more dilute bleach liquor.

The bleaching of cotton goods containing coloured stripes or other effect threads is always a delicate matter, for once the colour has been destroyed it is impossible to restore it, and the inevitable claims for damage then remain to be met. The essential point to notice in bleaching processes of this character is that various dyes are susceptible to bleaching liquors according to their pH. Some dyes are easily destroyed under acid conditions whereas they would be quite unaffected in slightly alkaline bleach liquors. For instance, cotton dyed with Turkey Red is fairly easily stripped in bleach liquors slightly acid (pH less than 7) whereas it is almost unaffected by a similar liquor whose pH slightly exceeds 7. Aniline Black dyed stripes are also best kept intact by the use of a slightly alkaline bleaching liquor.

In large scale practice it is usual to test samples of coloured materials in the laboratory before undertaking the bleaching of the bulk of the material. All such tests should be carried out with bleaching liquors of known pH and this same pH maintained in bleaching subsequently on a large scale. Also, in the case of colours which appear to be stripped it is advisable before deciding that the goods cannot be bleached to experiment with bleach liquors of various pH; under certain circumstances it may ultimately be found that the colours will withstand bleaching under conditions more alkaline than usual.

The successful bleaching of cotton goods is intimately connected with the control of the pH of the bleach liquors. This is therefore a subject of concern to all practical bleachers.

#### Colour in Textile Fabrics

"THE ways and means of textile colouration" were explained in a lecture delivered on Saturday to the members of the British Association of Managers of Textile Works, at the Manchester Athenaeum, by Mr. Ronald Humphries, who spoke of the work done by the Manchester chemist, Thomas Henry.

Superficially it would appear, said the lecturer, that the coming of the aniline colours, and the enormous number of these dyes research had revealed—the total was about 2,200, of which 1,500 were cotton dyes—would make the dyers' task to-day an easy one. The reverse, however, was the case, for each individual colour, even of the same class, had different dyeing fastness properties, and often required special treatment in application. The extent to which the dyer had succeeded could be judged every day in their homes and the wearing apparel of the people.

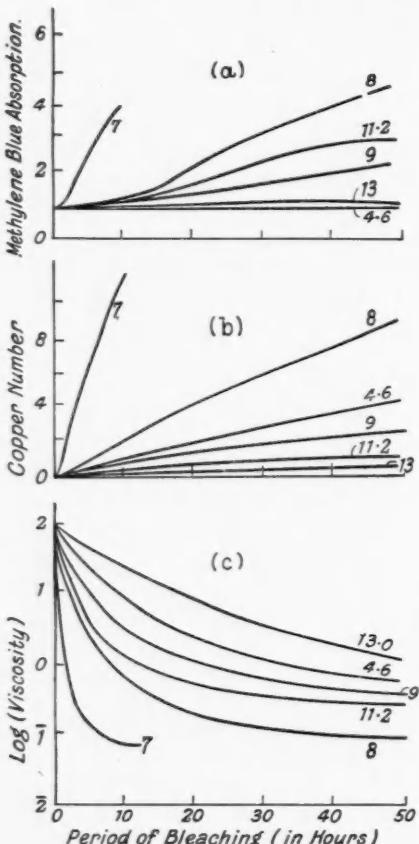


FIG. 2.—NUMBERS AGAINST CURVES DENOTE pH.

as 0.005. The Copper Number is not an exact measure of the strength of cotton, but in general the higher this number the lower is the tensile strength of the cotton. Also the higher the Copper Number the more likely is the white cotton to after-yellow during storage. A series of experiments was therefore conducted in which bleaching liquors of various pH were circulated through samples of cotton and from time to time small portions of the cotton were withdrawn and their Copper Numbers determined. The results are shown in Fig. 2b.

In these curves it is again very evident that the Copper Number is most influenced when the bleaching is carried out under exactly neutral conditions—the curve for pH=7 is exceptionally steep compared with the others.

It is generally known in dyeing practice that over-bleached cotton has a markedly increased affinity for basic dyes such as Methylene Blue. So that the Methylene Blue absorption of a bleached cotton can be regarded as indicating its degree of oxidation or deterioration. The curves for Methylene Blue

## Problems of "Dyeing and Colour Fastness"

By J. Guilfoyle Williams

The following is a summary of the address of Mr. J. Guilfoyle Williams, B.Sc. (London), to the students at the Drapers' Chamber of Trade Summer School at Girton College, Cambridge. The art and craftsmanship of the dyer, he said, was concerned with the production of colour effects and designs, but there was another aspect, not sufficiently studied, namely, the "science" of dyeing, which should very largely be concerned with the fastness of the colour effects secured.

THE first test in a colour was fastness to rubbing. Mr. Williams said, and this was performed by rubbing the colour vigorously with a white cloth. The test had two sections—dry rubbing and wet rubbing. Fabrics with colour loose to dry rubbing were usually very objectionable, as other fabrics, or the skin, would easily become stained, and even looseness to wet rubbing was often a serious objection. Many people believed that loose dye was dangerous because it might poison the skin, and, more particularly, that it was especially dangerous if the skin was broken. That belief was not founded on any true experience.

The next consideration was fastness to water. Colour loose to wet rubbing might be found to be appreciably extracted by cold water. Such cases had been met in umbrella covers which had shed coloured rainwater in a storm and discoloured hats and coats. Defective umbrella covers of that type were fortunately rare. But the defect might be met in some print silks, and it was not rare to receive complaints of water-loose colour in wool bathing costumes. Some dyestuffs were affected by water so that any drops spilled on the fabric would extract colour or would give a dark edge round the water stain. Most fabrics during their existence were exposed occasionally or frequently to washing.

### Recommended Tests

Sensible people usually washed coloured fabrics under the mildest possible conditions. But, on the other hand, if fabrics were sold as washing fabrics they should withstand even a rather severe ordinary washing, and if they were sold as "fast" or "fadeless" they should withstand severe treatment. It was a very simple test to wash a sample of fabric, and it was certain that if that was done many disappointments would be eliminated. There were far too many fabrics that fell down seriously in washing. If possible, a laboratory test should always be made, because colours might be satisfactory in a trial wash and fail in use. Other important points in testing washing fastness were colour-bleeding (or running) and colour marking off. In testing the colour fastness to washing a portion of similar white material was sewn on to the test piece. Some dyes which were extracted in washing did not cause staining on the white test piece, but in other cases staining, slight or serious, would occur. The degree of that bleeding must be considered in gauging the washing fastness. Dyestuffs were very complicated chemical compounds, and in many cases the colour was affected by hot-ironing. Sometimes the colour change was only temporary and after a short time reverted to its original shade. But occasionally it was permanent. There was rather a difficulty in setting a standard because an iron might be almost any temperature and, of course, a scorching temperature would spoil the fabric and nearly always affect the colour. But if the colour was affected by hot-ironing while it was damp, then it was certainly defective.

A point of some importance in regard to hot-ironing was the transfer of colour on hot-ironing. For instance, in a print frock one was almost bound to iron one part of the fabric while it was in contact with another part of the fabric. If colour transferred from the first part to the second part, then the fabric was spoiled. Test must, therefore, be made by hot ironing the fabric against white fabric first, when both were dry, and then when both were damp.

The subject of washing led to that of laundering. In certain cases, to secure good whiteness laundries had to use bleaching agents. Naturally they were not used on ordinary coloured fabrics, but in the case of shirtings, handkerchiefs, tablecloths and towels, bleach might be used. Colours that were perfectly fast to soap and soda might not be fast to chlorine bleach (chloride of lime or bleaching soda), and so on those items one tested the colours not only for fastness to light, perspiration, washing, alkali and hot-ironing, but also to chlorine bleaching.

### Fastness to Light

In regard to sunlight it was important to note that conditions of exposure had a great effect. It was an established fact that some coloured fabrics might fade more rapidly in Manchester than in the tropics. The essential factor in the fading was sunlight, but a very important auxiliary factor in many cases was the moisture of the fabric—and so in moist climates (and, of course, particularly by the sea) fading occurred much more rapidly. That, by the way, was probably the meaning of the "sea and air" part of the usual claims of "fast to sun, sea and air." The sunlight which caused colour fading was not simply direct sunshine and the ultra-violet light. The ordinary bright diffused daylight played a very large part in fading, and when tests were made exposing samples uncovered, covered with ordinary window-glass, and covered with Vita glass, it was found in many cases that the fading was about equal in all cases. Usually light fading involved loss of colour, but on occasion a darkening of colour might occur. In ordinary cases of fading the loss of colour was continuous and proportional to the exposure. With some vat-dyeings a slight loss of colour might occur after a short exposure, but that loss did not increase on further exposure.

In testing the fastness of colour it was therefore not sufficient to take the time to first visible fading, but the effect of further exposure must also be considered. The testing of light-fastness could either be done by actual exposure to sunlight or to artificial sunlight (ultra violet light apparatus could be used). For practical testing artificial light was essential. The total exposure given in the ultra violet light apparatus was one week if it was necessary to make sure the colours were grade 1. The fastness of dyeings to sunlight was sometimes very considerably affected by after-treatments.

Many people believed that certain colours were always fugitive to light. That was not a fact, for the stability of a dyestuff to light was a question of its chemical structure and not of its colour. Probably the belief arose from the experience of previous decades. For instance, mauve was generally supposed to be fugitive (though the author had not found this so) and it might be that belief dated from Perkin's mauveine, which was not a fast dye. The colour that was most often fugitive was a bright blue, though one could get that of excellent fastness. Red and yellow were generally of fair fastness to light. In compound shades produced by using several dyes, one might get colour change owing to one dye fading more rapidly than the others. Green was a colour frequently found to be fugitive and this might be due to the use of a fugitive blue.

### Relative Fastness

A familiar excuse in the case of colour failure in wear or wash was that no fadeless colours could be obtained. That was undoubtedly true, for a fadeless colour should withstand eternity. But it was begging the question, for fabrics would not last for ever, and the requirements of the most exacting customer were quite limited when expressed in the term of years. There were dyeings which would fade considerably after one day's exposure to light, and dyeings which would not fade much after five hundred days' exposure to sunlight. The second could be regarded as fadeless to washing.

From the consumer's and the retailer's point of view dyeings could be grouped into four broad classes. The factors which had to be considered in that classification would depend on the use for which the fabric was intended. In furnishings, the first requirement was clearly fastness to light. Rugs, carpets and to some extent furniture coverings, principally required fastness to light. But window hangings, etc., in addition to fastness to light, must have some reasonable fastness to washing as well. In actual testing, they made tests for fastness to light, weathering, washing and hot-ironing. Dress fabrics and suitings should have a good fastness to light. Generally speaking, men's suitings were reasonable in that quality—but

in women's dress fabrics the dyeings were usually acid dyeings or direct cotton dyeings, and a proportion were very fugitive to light.

A second requirement was fastness to perspiration, more particularly in dress fabrics. Fabrics sold for frocks could be divided into two classes—those for ordinary day wear and those for more particular occasions such as evening wear. In both cases one must preferably have fastness to perspiration—though in all cases where possible, shields should be worn. In exceptional cases, fabrics might be of a type that could not be washed or were not intended for washing—for instance, marocain, which shrank severely if washed, but could safely be dry-cleaned. But, in general, colours on fabrics for frocks must be reasonably fast to washing. And day wear fabrics must be of fair fastness to light. In underwear, colours should be tested for fastness to washing and to perspiration. Towels, handkerchiefs, and table linen should be fast to washing, and in a general case the dye should be very fast, as washing to secure stain removal might be severe or even chemicals might require to be used. Shirtings might be fast to light, as the cuffs and V-neck portions got considerable exposure, and very fast to washing, as they frequently got a very hot or boiling wash, and they should also be fast to chlorine, or, as already stated, trouble might occur in laundering.

#### Characteristics of Light Fading

Fading that was due to light had two characteristics. One face of the fabrics would usually be more faded than the other, and any covered portions would have sharply defined areas of little or no fading. The time of wear and the type of weather over the period must be considered in assessing the degree of blame attached to the fabric, for ordinary goods were certainly not fadeless. In ordinary dyeings bright blues and greens were often fugitive and basic dyes on silks were bright but fugitive. Basic dyes were usually used on straw hats, and in fact were necessarily used in some cases because they were the only ones which would penetrate; in consequence, colours here might be very fugitive. About 5 per cent. of women's wool dress fabrics were unfit for sale for outdoor wear on account of low light fastness, and with silks the proportion was probably at least 15 per cent.

In the case of complaints against fabrics known to be satisfactory in normal use, one might suspect hot-ironing effects. Careful inspection of the article would often give evidence where such damage had occurred. The shape of the iron might show, or more frequently it would be found that where two thicknesses of cloth overlapped, the protected portion was unaffected. Care must be taken to ascertain that such colour change was not due to sunlight fading. In one case where the author complained about "fadeless" casement, the actual cause of the fading was hot-ironing. When fabrics were soiled, the colours dulled and the appearance produced might be similar to sunlight fading. In a case of complaint against a carpet for fading, the cleansing of a small portion showed that no colour loss had occurred. When complaints of staining from fabrics were concerned and the colour was loose to dry rubbing, no defence of the dyeing was possible, but if the colour was only slightly loose to wet rubbing, then the dyeing was not good, but one could not class it as bad. In all cases of staining of the skin it was important to realise that the colour was not dangerous to health.

#### German Coal Tar Position

ACCORDING to a report of the I.G. Farbenindustrie for the quarter ending June 30, 1930, retrenchments are being effected in all industries producing coal tar and its products. Production of coke has decreased causing a shrinkage in the supply of crude tar. These retrenchments have alleviated somewhat the severity of the situation and explain the stationary position of crude tar prices. Coal tar products move sluggishly at unchanged prices. Tar roofing finds no outlet owing to a very slight volume of building. Tar for road paving still awaits fulfilment of the Government's plans for extensive road-building projects. Pitch from coal tar was in decreased demand, but recent inquiries for large lots from foreign countries have brightened the outlook. Prices are stationary, but cuts are awaited if the market situation shows no early change for the better. The position of distilled and prepared tar has improved somewhat. Coal tar oils move comparatively well at satisfactory prices. Anthracene oil is in oversupply at reduced prices.

## Dyes and Textiles

### Notes on Current Researches

*Quantitative test for damaged wool.*—A long-felt want for a convenient and accurate means for quantitatively estimating "soundness" of wool appears to be met by a method recently described by C. Rimington<sup>1</sup> (of the Biochemical Department of the British Research Association for the Woolen and Worsted Industries). The earlier methods of Pauly and Binz<sup>2</sup>, and of W. Sieber<sup>3</sup> are useful but more of a qualitative character only.

In this new quantitative test, use is made of the Pauly method to a limited extent, since it is based on the fact that the cortical portion of the wool fibre freely reacts with a solution of diazotised sulphanilic acid whereas the epithelial scales are inert. These scales in an undamaged wool fibre so protect the cortex that it cannot react with the diazo solution, but when the fibre is damaged and the scales disturbed or partially removed the cortex becomes sufficiently exposed to react and exhibit colour. Presumably the brownish red colour is an azo compound formed by coupling of the diazotised sulphanilic acid with the tyrosine in the cortex.

Rimington's modification of the Pauly test consists of dissolving out the colour and matching the intensity of the solution against a standard solution of the dye, New Acid Brown S (B.D.C.). By selecting arbitrary units, it is thus possible to construct a scale by means of which the extent of damage of any given sample of wool can be expressed numerically. Details of the test are as follows:—

The reagent is prepared by mixing 10 c.c. of a 10 per cent. solution of sodium sulphanilate with 5 c.c. of an 8 per cent. solution of sodium nitrite, then adding 2 c.c. of concentrated hydrochloric acid down the side of the vessel, mixing with a gentle motion and allowing to stand for one minute before use. A suitable quantity of the wool to be tested (about 0.1 g.) is weighed and then wetted out in 15 c.c. of a 9 per cent. solution of sodium carbonate; to this the reagent is added. After exactly 10 minutes the wool is withdrawn, rinsed thoroughly in water, and transferred to a test-tube, 4 c.c. of a 10 per cent. solution of sodium hydroxide added, the tube placed in a bath of boiling water for exactly five minutes, and the resulting reddish solution transferred quantitatively to a graduated volumetric flask and made up to a volume of 5 to 25 c.c. (according to the depth of colour). This solution is then matched against a 0.1 per cent. solution of New Acid Brown S, using a Dubosc or Kober colorimeter if more than the usual works' degree of accuracy is desired.

Unfortunately the method is obviously not applicable to dyed or naturally coloured wool materials.

*The reactivity of silk fibroin.*—A real silk fibre consists of the true filament of fibroin and an outer covering of the gum sericin, this latter being readily removed by treatment with a boiling soap solution. The fibroin contains about 18 per cent. of nitrogen in the amide form, but by hydrolysis with alkali or acid it can be converted into amino nitrogen which can be readily determined. In some recent investigations<sup>4</sup>, M. Harris and T. B. Johnson have dispersed pure fibroin by various methods and observed the corresponding changes in the amino-nitrogen content.

Fibroin was dispersed by treatment with a 50 per cent. lithium bromide or a 70 per cent. calcium thiocyanate solution, and also by mechanical means such as by grinding in a pebble mill. These processes produce different results for fibroin dispersed with the aid of a concentrated salt solution had an amino-nitrogen content of 4.9 per cent. (before dispersion this amino-nitrogen content was negligible) and the mechanically dispersed fibroin contained not more than 0.5 per cent.

A direct result of the increase of amino-nitrogen content is that the dispersed fibroin acquires a new susceptibility to attack by bacteria, and also changes its levo optical rotation to a dextro rotation. The mechanically dispersed fibroin was considerably less attacked by trypsin than the fibroin dispersed by salt solutions. It would thus seem advisable in commercial degumming and other treatments of silk materials to avoid dispersion of the silk fibroin if the durability of the yarn or fabric is to be maintained at its maximum.

<sup>1</sup> *J. Text. Inst.*, 1930, **21**, 237 T.

<sup>2</sup> *Zeit. für Farben- und Textilind.*, 1904, **3**, 373.

<sup>3</sup> *Textilberichte*, 1928, **9**, 326.

<sup>4</sup> *Ind. and Eng. Chem.*, 1930, **22**, 905.

**Dyes and Their Application : Recent Technical Progress**

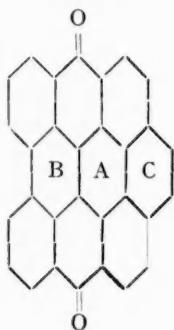
By L. J. Hooley

**Vat Dyes**

If we exclude for convenience the perylene and pyrene classes, a large proportion of post-war results in the remaining branches of anthraquinone vat chemistry are seen to consist of the production of new vat dyestuffs from old ones by adding substituents. In some instances an old product, useless because lacking in affinity or other tinctorial property, has been slightly modified in structure and the disadvantage removed. Some of these developments will be briefly dealt with below.

**Dianthrone**

The yellow vat dyes *ms*-benzodianthrone (helianthrone) and *ms*-naphthodianthrone are cases in point of dyes lacking affinity; the former is represented by the formula shown,

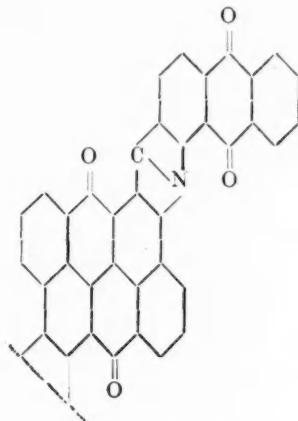


but has only one ring in the molecule (A), and the latter by the same with the A and B rings. The addition of another *ms* ring (C) gives the two new bodies *allo-ms*-naphthodianthrone (B and C) and anthradianthrone (A, B and C), which have increased affinity. These and several of their derivatives have recently been described. Of these derivatives the halogens are of special interest, firstly because the halogenated dyes themselves are new and often give brighter and deeper shades than the originals, and secondly because the halogens can be replaced by heavier groups with still greater changes in shade. Methods for halogenation can be found in G.P. Appln. 30,025, while B.P. 303,095 describes more complex derivatives obtained by replacing the halogens (or other negative substituents) by treating with bodies containing replaceable hydrogen atoms such as alcohols, phenols (dichloro-*allo-ms*-naphthodianthrone with phenol gives a blue-red) and amines, especially aminoanthraquinone (B.P. 307,364)—I.G.F.A.-G.

**Anthranthrones**

Closely parallel results are to be seen with anthranthrone. Although this was discovered by Kalb (in 1912), see D.R.P. 280,787 (2614/13), it is only during these last two or three years that it has been further investigated. Again halogenation and the replacement of halogen atoms may be noticed. Apart from direct halogenation, chlorine or bromine may be introduced *via* the sulphonic acids (Cassella, B.P. 304,613). The halogen anthranthrones are converted to sulphates in acid of 80-95 per cent. strength: these are probably oxonium salts, and can be used for purification, separating as violet-black crystalline salts. (B.P. 316,149, I.G.F.A.-G.)

As examples of the utilisation of the halogen substituents, there may be cited their replacement by cyano groups by boiling with cuprous cyanide in quinoline or benzyl cyanide (B.P. 307,481), also their replacement by sulphur groups by treating with compounds such as potassium xanthate (2 : 7-dibromoanthranthrone gives a reddish-violet, probably 2 : 2'-dibromo-7 : 7'-dianthanthonylthioether) (B.P. 305,489). Further, more complex derivatives may be used such as aminoanthraquinones (B.P. 286,669), 1-aminoanthraquinone and the dibromoanthranthrone from 4 : 4'-dibromo-1 : 1'-dinaphthyl-8 : 8'-dicarboxylic acid gives a grey-blue. The use of 1-aminoanthraquinone-2-aldehyde gives a brown, which is probably an acridine (B.P. 296,373)—I.G.F.A.-G.

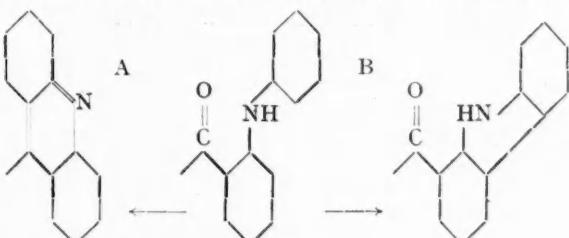
**Pyranthrone**

Somewhat similar derivatives are to be noted in the case of pyranthrone as with the various anthrone dyestuffs already mentioned. The halogenation of this body is old, and halogenated products are on the market, but some new variations are seen, as in B.P. 321,161, where more than 4 atoms are introduced by halogenating in chlorosulphonic acid with a catalyst such as iodine or selenium; also in B.P. 320,168 (I.G.F.A.-G.) when a product which is stated to be practically exclusively the dibromo is obtained by brominating in water; and neutralising the hydrobromic acid as quickly as formed, also in B.P. 306,434 (Brit. Aliz. Co., and P. Beghin), where brown vat dyes result from heating pyranthrone with sulphuryl chloride in nitrobenzene at 90-140° C. These and other halogenated pyranthrone can be then condensed with aminoanthraquinones.

**Carbazoles**

Bodies containing two anthraquinone molecules forming two sides of a pyrrol ring have been obtained by the condensation of phthalic anhydride with carbazole. The product from carbazole itself is a yellow which is not very satisfactory, but from the corresponding N-ethyl-carbazole the well-known Hydron Yellow G is obtained. Dyes which have been understood to have similar structure have also been obtained by treating dianthrimides with condensing agents such as potash or aluminium chloride. Recently new variations of this latter synthesis have been described, both by selecting particular starting materials and by varying the methods of synthesis or purifications. Thus in B.P. 296,758 the products from 1 : 5- or 1 : 8-di-( $\alpha$ -anthraquinonylamine) anthraquinones have been brightened by treating with concentrated sulphuric acid below 5° C., pouring on ice, washing acid free and oxidising with hypochlorite at 70-80° C.

The cyclisation which takes place when dibenzoyldiamino-1 : 1'-dianthrimides are treated with sulphuric acid is now stated to be probably the same type giving a carbazole (B) instead of an acridine-(A) as originally generally assumed.



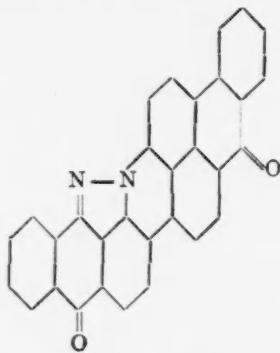
B.P. 298,666 describes the production of some new dibenzoyldiamino-1 : 1'-dianthrimides substituted by alkoxy or hydroxyl groups as starting points for this reaction. In B.P. 305,082 similar ring-closure is effected with dianthraquinonyl-amines, having an acridone ring and an aroylaminio ring.

Where the benzoylamino groups are in the 4 and 5<sup>1</sup> positions saponification can be carried out under mild conditions to remove the 5<sup>1</sup> acyl group only, the resulting amine giving reddish shades (B.P. 307,306)—I.G.F.A.-G.

#### Thiazoles, Pyrazoles

Speaking generally, anthraquinone vat dyestuffs containing four-membered heterocyclic rings have not been found of much importance. A number of pyrazol, thiazole and oxazole derivatives have, however, been described, and of these the pyrazoles have attracted most attention because of the dipyrizolanthrones obtained on fusion. Latterly one or two more derivatives have been mentioned such as the condensation products of acid chlorides of anthrathiazoles (obtained by heating 1-chloroanthraquinone-2-carboxylic acid with sodium polysulphide and ammonia, and then converting to the acid chloride) with aminoanthraquinones (Du Pont U.S.P. 1,705,023 and 1,706,981).

The acidic -NH<sub>2</sub> group in 1:9-pyrazolanthrone, in addition to being alkylated or arylated, may be treated with substances such as o-bromobenzoic acid, o-chloronaphthoic acid and chlor-benzanthrone (B.P. 298,775, I.G.F.A.-G.), and in suitable cases further ring-closure may be effected as in B.P. 298,284 (I.G.F.A.-G.) to give products such as the following.



Indanthrone

These are always of interest, as the best of the vat blues. In general they do not seem to lend themselves well to substitution, except by halogen, as the shade or other tinctorial properties suffer in consequence. New details for manufacture and purification appear occasionally, such as in B.P. 320,397 (I.G.F.A.-G.), where purification is by heating with manganese dioxide in sulphuric acid at 30-70°C. As another method of purification the alkaline melt may be diluted with water to about 3-4 per cent. KOH, a little hydrosulphite added and the alkali salt of the leuco-indanthrone allowed to crystallise out (U.S.P. 1,679,230 Nat. Anil. Co. and D. G. Rogers).

The already known products obtained by treating indanthrone with formaldehyde are converted to new greenish-blue vat dyestuffs by oxidation, with, for example, bichromate (B.P. 322,281). New products are obtained from halogen-indanthrones by treatment with cuprous cyanide—these also dyeing in greenish-blue shades (B.P. 309,192) I.G.F.A.-G.).

### Dyestuffs Markets Review

#### Lancashire

It is not a little remarkable that during a period when the whole of the textile industry is reported as being in an extremely depressed condition, sales of dyestuffs should be maintained at a relatively high percentage of the normal. It does appear to be the case that very much less short time is being worked in the dye-making industry than in the spinning and weaving sections of the trade.

It is, of course, true that, so far as the cotton industry is concerned, the grey and bleached section of the industry has been very much harder hit than the coloured, but this does not seem to be a sufficient explanation of the anomaly mentioned. No doubt a partial explanation is that the use of vat dyes continues to increase, and these dyes, requiring more elaborate processes of manufacture in many cases than the

direct and acid dyes, tend to keep larger numbers of men employed. Whatever the real explanation may be, the fact remains that the dyestuff manufacturers are affected much less seriously than those engaged in the purely textile division of the industry.

Although no official information is available, the feeling is growing, amongst dye-users at least, that the Government will not prolong the period of protection for the dyestuff industry. There are dyers and printers who, whilst feeling that it is unfair to expect the textile industry to stand the consequences of higher dyestuff prices here than abroad, nevertheless believe that the maintenance of the manufacture of synthetic dyestuffs on a large scale in this country is necessary to the well-being of the State. The influence of the existence of such a highly scientific industry on other branches cannot easily be overlooked. The dyer and printer realise that improvements in dyeing and printing processes are mainly the work not of the dyer and printer, but of the dyestuff manufacturer.

The existence of a large number of chemists trained in this kind of work has most certainly the effect of producing a higher scientific standard in all industries which depend upon chemistry in any degree, and many dye-users in these days would be prepared to support a well-devised scheme of carefully regulated subsidies for dyestuff manufacture for a few more years. In a sentence, the objection is not primarily to some measure of protection, but to that kind of protection which makes the textile industry pay for the benefit that the community as a whole receives.

#### Scotland

As the end of the year gets closer, interest centres in the fate of the Dyestuff Act to the exclusion of minor matters. All the important bodies, whether holding a direct or indirect interest in the industry, have now had their say on the matter, but as to what is to be the outcome no one is yet any the wiser. It would be difficult to imagine a less favourable time for change, which will increase the difficulties of industry. In addition, the uncertainty of the position is bound to have some adverse effect on business already.

As regards more detailed matters, November has been much the same as October. The tweed trade is managing to hold its own and locally some manufacturers are fairly busy, and although others are very short of orders it is a satisfactory feature, that on balance trade is being maintained. Dyeing and printing have been very quiet and dyestuff and chemicals sales fallen a little below last month.

#### Swiss Dye Exports in 1930

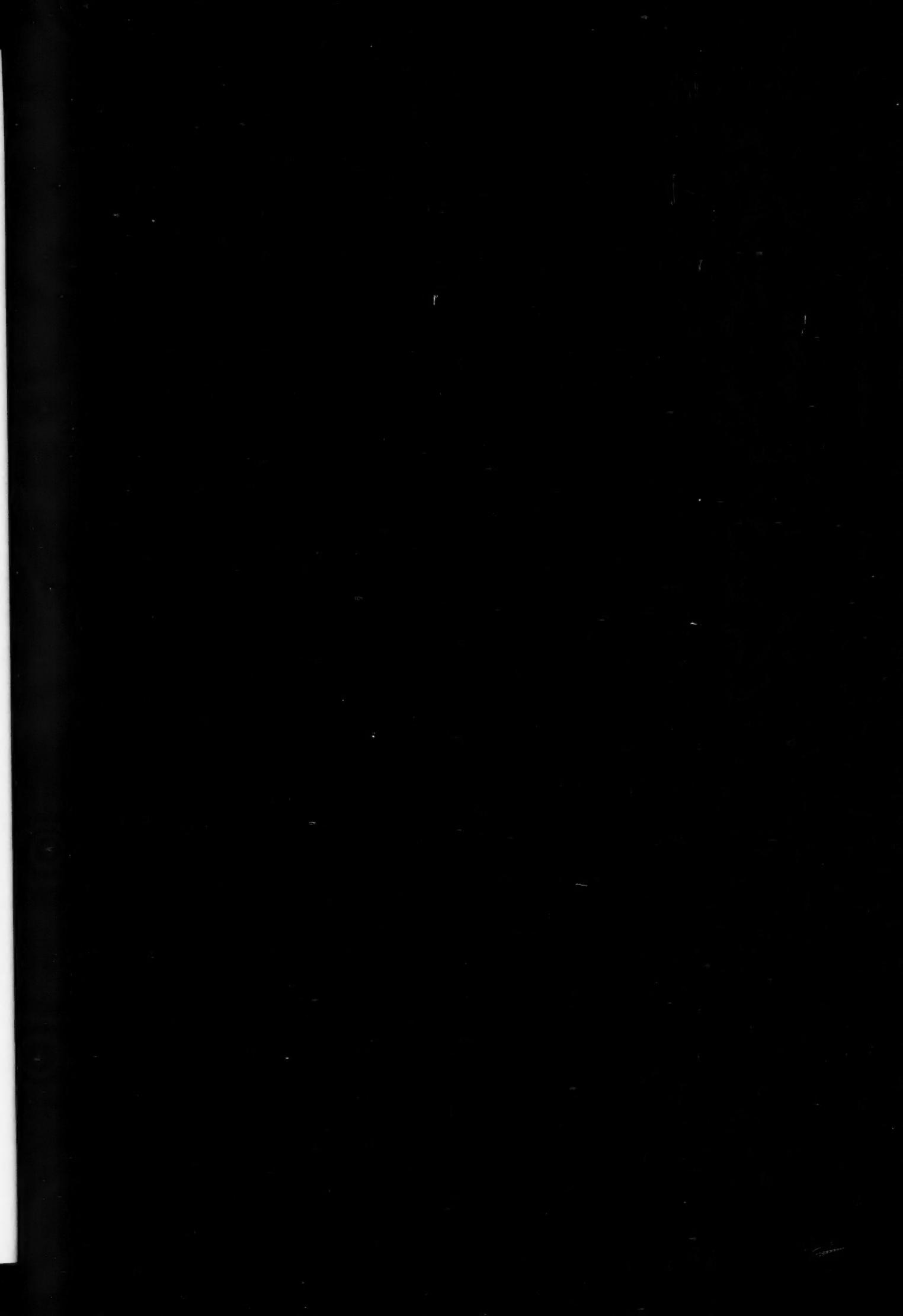
DURING the first eight months of this year, Swiss dye exports fell in value by over £250,000 from the first eight months of 1929. The distribution of exports among the principal markets in 1930 was as follows :—

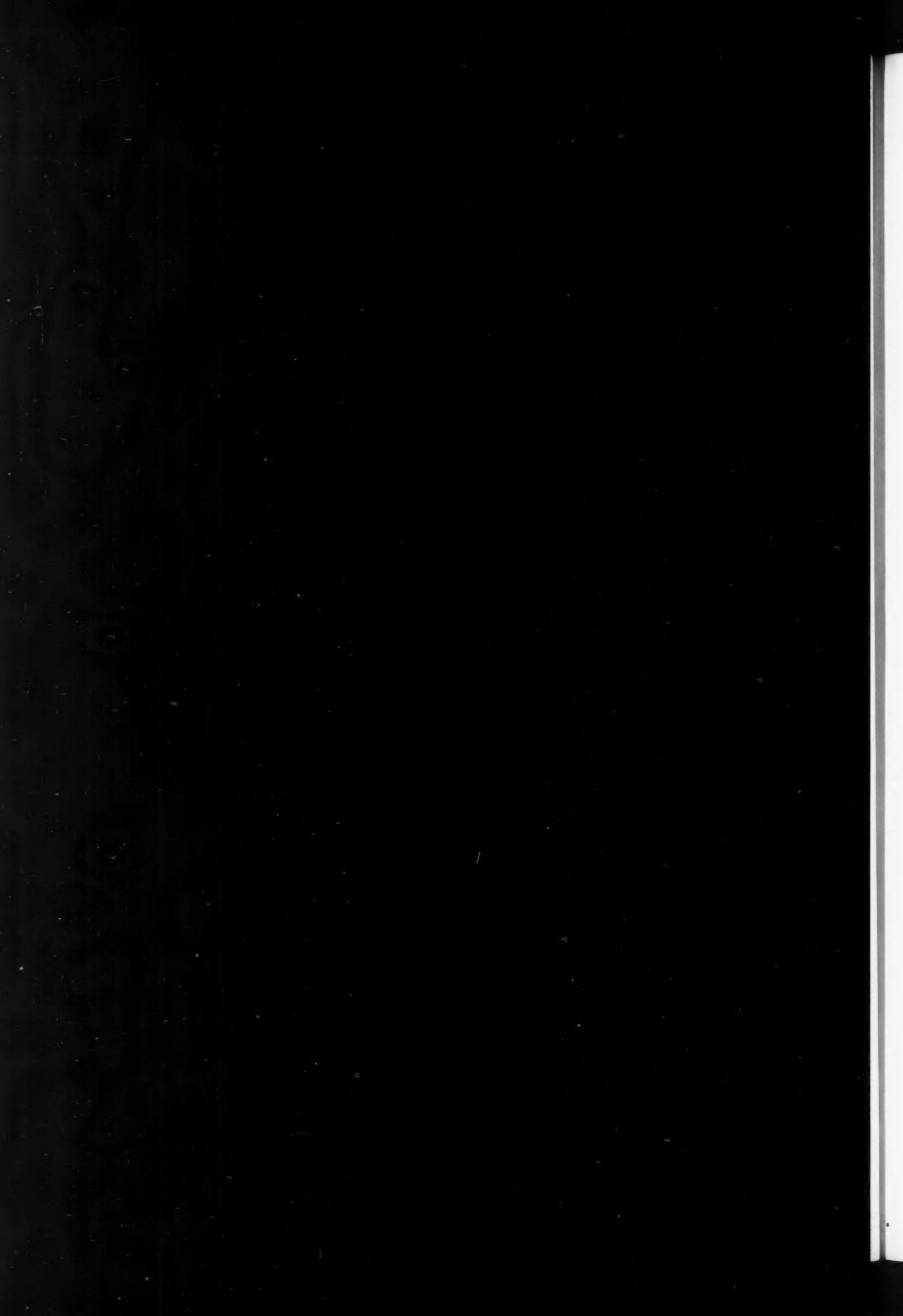
##### ANILINE AND OTHER COAL-TAR COLOURS.

| Destination.                       | Metric Quintals. |
|------------------------------------|------------------|
| Germany .....                      | 10,025           |
| Great Britain .....                | 4,739            |
| France .....                       | 3,876            |
| United States .....                | 3,306            |
| Italy .....                        | 2,652            |
| Czechoslovakia .....               | 2,985            |
| Japan .....                        | 1,409            |
| Belgium .....                      | 2,383            |
| China .....                        | 1,693            |
| Other .....                        | 13,425           |
| Total: 1st eight months 1930 ..... | 46,493           |
| Total: 1st eight months 1929 ..... | 52,517           |

##### INDIGO AND SOLUTION.

| Destination.                | Metric Quintals. |
|-----------------------------|------------------|
| China .....                 | 8,079            |
| British India .....         | 837              |
| Japan .....                 | 594              |
| Other .....                 | 659              |
| 1st eight months 1930 ..... | 10,169           |
| 1st eight months 1929 ..... | 11,957           |





# Dyestuffs Monthly Supplement

Published in the second issue of "The Chemical Age" each month

Communications relating to editorial matter for the Dyestuffs Monthly Supplement should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Advertisement matter, subscriptions, etc., should be sent to the Manager. The Supplement is devoted to the interests of both manufacturers and users of dyestuffs, and contributions will be welcomed.

## New Year Spring Shades

Imperial Chemical Industries, Ltd., issue two attractive folders, showing ten colour combinations suitable for the production of the fashionable shades for spring, 1931. They are described as "only a few suggestions," and recipes will readily be supplied for production of any other desired shade on any type of material. The selected colours illustrated are Honeycomb, Flamingo, Cactus, Burnt Almond, Lagoon Blue, Hyacinth Blue, Topaz, Prunella, Willow and Ruby. One folder shows these shades on silk and artificial silks, where the colours are distinguished for their brilliant clearness; the other illustrates their application to wool, where the effects are naturally warmer and softer, but still full of colour character.

## Chlorazol Colour Album

One example of the splendid work that the British Dyestuffs Corporation has done for the British textile industry is supplied by the magnificent album of Chlorazol Colours for cotton fabrics, just issued by Imperial Chemical Industries. The range now extends to 141 distinct shades, shown in different strengths, and constituting a very beautiful collection. Complete fastness tables are published for all these colours, with full instructions for dyeing, after-treatment, etc. The Chlorazol colours are divided into groups according to their suitability for acetate silk resist work, for dyeing cotton effects (curl dyeing) in woollen piece goods, for working in conjunction with size, for covering unripe cotton (neps), and for withstanding mercerising.

Every precaution, it is stated, has been taken to make the classification in the fastness tables as accurate as possible, but it is pointed out that since it is impossible to test colours for fastness under all the variations of conditions which obtain in practice, the value of the table is limited to service as a guide to the dyer in the search for dyestuffs to meet his particular need. In view of the lack of common agreement on standards of fastness, customers are recommended, in those special cases in which a particular fastness property is of prime importance, to make tests on their own material under their own conditions.

## Fast Colours for Cotton

In addition to the general album of Chlorazol Colours for Cotton, Imperial Chemical Industries publish a separate illustrated list of Chlorazol fast colours applied to cotton piece goods. The colours shown number 48, and each colour is illustrated in three strengths, showing a fairly wide range of shade. The products included in this card are chosen from the Chlorazol range on account of their superior fastness to light. At the same time, it is stated, in the production of combination shades it is not sufficient merely to choose component dyestuffs individually possessed of good fastness to light, but the nature of the "fade" should also be taken into consideration. For this reason the manufacturers have confidence in recommending, for browns and drabs, a combination of Chlorazol Fast Orange AGS, Chlorazol Brown MS and Chlorazol Fast Blue 2BNS, which, in practice, has been found to yield dyeings which retain their tone on exposure.

The methods of application and of after treatment are very much the same as in the case of the other colours.

## A New Chlorazol Colour

Together with the albums noticed above, Imperial Chemical Industries announce the production of a new Chlorazol colour Fast Orange WS, which is an addition to the existing range of direct cotton colours. Chlorazol Fast Orange WS possesses fastness properties similar to those of Chlorazol Fast Orange RS, but has the advantage of superior solubility. It is suitable for dyeing all types of cotton materials, particularly cops, beam warps, etc., in circulating machines. It gives a bright shade of reddish orange possessing good all-round fastness which, combined with good levelling and general dyeing properties, makes it of special interest for dyeing cotton piece goods. This colour possesses only slight affinity for wool or silk and is suitable for covering the cotton in union materials when applied at low temperatures. It is also suitable for dyeing silk, artificial silk, jute, tagal, wood chip, and paper. It may be used for the direct printing of cotton, wool, or silk, and as a ground shade for white or coloured discharges with tin crystals.

## Dyestuffs Licences for November

The following statement relating to applications for licences under the Dyestuffs (Import Regulation) Act, 1920, made during November, has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee. The total number of applications received during the month was 483, of which 396 were from merchants or importers. To these should be added 10 cases outstanding on October 31, 1930, making a total for the month of 493. These were dealt with as follows: Granted, 460 (of which 444 were dealt with within seven days of receipt); referred to British makers of similar products, 18 (of which 13 were dealt with within seven days of receipt); outstanding on November 29, 15. Of the total of 493 applications received, 457, or 93 per cent., were dealt with within seven days of receipt.

## New American Colours

The Colour Co-ordination Committee of the U.S.A. National Retail Dry Goods Association have selected seven dress colours for the holiday and late winter season of 1931. They are Hilit Blue, Clematis Blue, Amulet Blue, Fraise, Woodland Green, Rose Cedar, and Muscade Beige. A resolution was unanimously passed by the committee requesting manufacturers to use the official names of the Textile Colour Card Association when showing these colours in their lines. In this way retail buyers may make out their orders in these names, with the assurance that manufacturers will easily identify the colours ordered.

In addition to the selection of seven dress colours for holiday and late winter selling, the committee also adopted a group of shades for southern resort wear. It was felt that dead white would be an outstanding factor in this field, especially with such colours as Admiralty (a clear navy blue), Wildfire (a medium yellowish red), Blue Jade (a vibrant green), Stargol (an animated yellow), and Indies Brown (a darker neutral tone).

In addition to white, alone or with "accent" shades, the following were selected as basic ensemble colours: Amulet Blue (a true turquoise), Flower Blue (a sky tone), Stargold, May Bud (a soft green of yellowish cast), and Seashell Pink (a pastel tone).

## Influence of Dyes on Durability of Dyed Materials

By "Colorist"

Owing to the increasing use of textile materials, particularly those of cotton, for purposes apart from clothing, it will be necessary to pay increased attention to the processes by which textiles suffer deterioration by exposure to light or attack by micro-organisms. In this article an account is given of the influence, protective or otherwise, exerted by dyeing processes or dyestuffs on the durability of cotton and wool fabrics.

MOST people accept the fact that clothes wear out with resignation and without criticism, and in these days, in marked contrast to the "good old days," there are few people who desire their garments to last the greater part of a lifetime. Yet textile materials are largely used for purposes other than as clothing, and there is much evidence to indicate that in the future fabrics will be equally important for utility purposes in the home and industry. Apart from its use in oilcloth and linoleum, tent material, upholstery, motor-car bodies and hoods, it appears likely that textile fabric will find use for home decoration in place of paper and wood panelling, and in engineering. Already, following success in America, the experiment is being made at Burnley of utilising cotton fabric for road construction. The durability of a fabric thus remains one of its most important properties.

A fabric of any material can suffer much damage as a result of stretching, twisting, and abrasion. But more subtle causes of deterioration are due to the action of light, and bacteria and mould fungi growth. These latter influences are the more effective because they are not often considered and, therefore, seldom retarded by preventative measures.

The action of light on a textile material, whether it be wool or cotton or rayon, is largely dependent on the presence of substances capable of rendering the light energy more or less effective. It is well-known that the energy of light varies with its wave-length, so that coloured substances, particularly dyes, can thus be very influential in determining whether or not the light incident on a textile material exerts its total energy content in destruction of the material. On the other hand, micro-organisms can usually have their effect in the absence of light; they merely require a certain limited amount of moisture and warmth. For its growth a bacillus or mould fungus requires food, and, in general, this must be largely obtained from the textile material on which it thrives. In yielding food to micro-organisms fabrics suffer disintegration and loss of durability.

### Mineral Khaki

The influence of dyes in textile materials exposed to the action of light may be protective or otherwise; much depends on the nature of the dyestuff. Thus it has long been observed that cotton fabrics dyed with mineral khaki withstand excellently use in tropical countries where the sunshine is very powerful. This dye is a mixture of the oxides or hydroxides of chromium and iron, and it has been established by P. W. Cunliffe and E. Midgley (*J. Text. Inst.*, 1928, **19**, 169) that of these two components the chromium oxide is much the more effective. The following data obtained by exposure of warp satin cloths (dyed and undyed) to sunlight and to the artificial sunlight from a carbon arc lamp illustrate this protective action very well:—

#### EXPOSURE TO SUNLIGHT (532 HOURS).

##### WORK OF RUPTURE OF FABRIC.

| Fabric.                      | Unexposed. | Exposed. | Tendering. | Per cent. |
|------------------------------|------------|----------|------------|-----------|
| Scoured fabric               | 31.8       | 13.4     | 58         |           |
| Scoured and dyed with chrome | 35.1       | 31.4     | 11         |           |
| " " " " khaki                | 38.1       | 32.0     | 16         |           |
| " " " " iron                 | 36.8       | 26.0     | 29         |           |

#### EXPOSURE TO CARBON ARC (12 HOURS).

##### Tendering.

##### Per cent.

|                              |    |
|------------------------------|----|
| Scoured fabric               | 45 |
| Scoured and dyed with chrome | 5  |
| " " " " khaki                | 3  |

The smaller protective influence of the iron as compared with that of the chrome is easily evident. As will be seen later, chrome also exerts a protective influence in the bacterial decay of wool.

Cunliffe and Midgley also found that organic dyes and the mordants employed for their fixation on cotton have a

mild protective effect. This would be expected in the case of basic dyes, for it is well known that tanned nets and sail cloth have a longer life than if not so treated. In dyeing basic dyes on cotton it is usual to employ as a mordant tannic acid and tartar emetic or other antimony salt. Cotton fabric mordanted according to industrial methods showed less tendency to lose its strength than non-mordanted fabric when exposed to light from a carbon arc, but in contrast to this the same mordanted material was more rapidly destroyed when weathered by exposure at Didsbury and on a voyage to Australia.

The peculiar difference between the tendering by artificial sunlight and outdoor exposure to sunlight (this includes weathering) was further shown by similarly mordanted fabric also dyed with the following basic dyes: Malachite Green, Safranine T, and Acridine Orange. In the case of Methylene Blue it appears that this particular basic dye has a special protective effect, for the dyed fabric was less tendered both with natural and artificial sunlight. Somewhat less conclusive experiments indicated that a few direct dyes showed a small protective action.

### Tests of Exposed Fabric

Unfortunately, organic synthetic dyes, for the most part, can also assist the destructive action of sunlight. This effect has been studied more particularly in the case of cotton dyed with various types of dyes. As might be expected, dyes which fade easily appear to have but little accelerating influence on the action of light on cotton: the dye is perhaps destroyed in preference to the fibre on which it is dyed. This effect is illustrated by some interesting fading results obtained about three years ago by Miss E. Hibbert (*J. Soc. Dyers and Col.*, 1927, **43**, 293). A piece of plain calico together with other pieces dyed with direct colours was exposed in Manchester for six months (660 hours) to sunlight until the colour was practically destroyed. The exposed samples were then carefully cleansed and their copper numbers determined with the following results:—

| Fabric.  | Copper Number. |
|--|----------------|
| Plain calico                                     | 0.6            |
| " .. exposed for 660 hours                       | 3.6            |
| " .. dyed with Brilliant Copper Blue and exposed | 3.8            |
| " .. " Chlorazol Sky Blue                        | 3.6            |
| " .. " Benzopurpurine 4B                         | 3.7            |

The copper number (a measure of the deterioration of the cotton) of the non-dyed sample is only slightly less than that of the dyed samples; the influence of the dyes thus appears to be negligible in either accelerating or retarding the action of sunlight. But when cotton dyed with much faster vat dyes was exposed to light (from a Fadeometer) the strength of the cotton in the faded material was shown to be dependent on the dye present thus:

| Dye on cotton fabric. | Strength of cotton. |    |
|-----------------------|---------------------|----|
| Before exposure.      | After exposure.     |    |
| lb.                   | lb.                 |    |
| Duranthrene Yellow    | 48                  | 27 |
| Cibanone Yellow       | 46                  | 16 |
| Indigo                | 46                  | 22 |

In the case of Duranthrene Yellow the dye appears to protect the cotton more than when Indigo or Cibanone Yellow is present.

Not only is the character of the dyestuff of importance as regards its catalytic influence; its state of oxidation is also important. This feature has been excellently made clear by some researches of F. Scholefield and C. K. Patel (*J. Soc. Dyers and Col.*, 1929, **45**, 175) using the leuco and fully oxidised forms of certain vat dyes. At the same time it was shown that the colour of the dye had definite significance.

In dyeing cotton with yellow or orange vat dyes, repeated lifting of the cotton from the dye bath so as to expose it to

light and air has the effect of tendering it. The light is directly concerned in this phenomenon since it appears to activate the leuco form of the vat dye and thus accelerate oxidation of the cotton. An interesting experiment serves to illustrate this point. Skeins of cotton yarn were dyed separately in portions of the same dye vat, one skein being exposed to sunlight and air periodically by lifting it out of the bath, the second being dyed and oxidised in a dark room, and the third immersed below the surface of the dye liquor during the whole of the dyeing operation, and finally washed in cold running water until oxidised. These three skeins were then tested as regards their strength, with the following results:—

| Treatment of skeins.                             | Strength. |
|--|-----------|
| Original yarn .....                              | 21.5 ozs. |
| Skein dyed and exposed to air in dark room ..... | 20.6 "    |
| Skein dyed and washed without exposure .....     | 21.1 "    |
| Skein dyed and exposed to light and air .....    | 18.9 "    |

Viscose rayon yarn is particularly sensitive to the destructive action of light under such dyeing conditions. The following results obtained by exposing this type of rayon during dyeing to light from a Fadometer show that the type of dye has an important influence:—

|                                   | Strength     |          |
|-----------------------------------|--------------|----------|
|                                   | Not exposed. | Exposed. |
| Cibanon Orange R .....            | 242.8 g.     | 101.2 g. |
| Cibanon Yellow R .....            | 229.5        | 88.3     |
| Chloranthrene Gold Orange Y ..... | 231.5        | 99.8     |
| Indanthrene Gold Orange 3 .....   | 232.8        | 180.2    |
| Diamine Fast Yellow FF .....      | 228.8        | 228.8    |
| Chlorazol Fast Orange R .....     | 229.1        | 224.7    |
| Without dye .....                 | 230.2        | 227.0    |

The progressive nature of the deterioration of the cotton being dyed during exposure to sunlight was established by dyeing a strip of cotton fabric with Cibanone Orange R paste in the usual vat, then lifting it for exposure to light and air for 10 minutes, cutting off a portion, immersing the residue in the vat for a further 5 minutes, again exposing for 10 minutes, cutting off another portion, again immersing the residue and so on. The successive portions cut off were then tested for their Copper Number with the following results:—

| Copper Number.                   |      |
|----------------------------------|------|
| Original cotton fabric .....     | 0.2  |
| ditto dyed but not exposed ..... | 0.3  |
| ditto with one exposure .....    | 0.7  |
| ditto " two exposures .....      | 1.3  |
| ditto " three exposures .....    | 1.5  |
| ditto " four exposures .....     | 1.7  |
| ditto " five exposures .....     | 1.75 |

It is evident that the durability of dyed cotton materials can be appreciably affected by sunlight, both during and after dyeing. Before the investigations of Scholefield it was not generally suspected that light could have a deteriorating influence during the dyeing process itself.

It will now be interesting to turn to the influence of dyestuffs on the durability of textile materials attacked by bacteria and mould fungi. Rather more attention has been given to wool than cotton. Most of the investigations relating to cotton have been carried out with the purpose of protecting colourless sized yarn from mildew so that dyestuffs have not been seriously considered in this connection; only a number of spasmodic references are to be found concerning the anti-septic properties of dyestuffs, although it seems fairly certain that a number of dyes (because of their chemical constitution) must have the power to protect cotton materials from the attack of micro-organisms.

#### Protection to Wool

Several years ago R. Burgess (*J. Text. Inst.*, 1924, **15**, 581) found that many dyes showed no protection to wool, and among those were Helianthine C, Roccelline, Benzyl Green B, Victoria Violet 4BS, Acid Violet 6BN, Erioglaucine Supra, Cloth Fast Orange G, and Orange II. But more recently Burgess has found (*J. Text. Inst.*, 1930, **21**, 444) that the chrome mordant employed for dyeing wool with various dyes has a very definite protective influence against bacteria mildew, particularly *Aspergillus niger* and *Penicillium brevecale*.

In these recent investigations, pieces of worsted fabric were dyed with 131 different dyes, then inoculated with mould spores and kept for a prolonged period in large air-tight jars containing a quantity of water. The results indicated that 49 of the pieces showed a definite resistance to mildew, and

of these 46 had received a chrome treatment in the dyeing process. This discovery of the importance of chrome was followed up by a large number of experiments for the purpose of ascertaining in what manner the chrome exerted its protective influence. Generally the presence in wool of 0.5 per cent. of chrome, either as  $\text{CrO}_3$  or  $\text{Cr}_2\text{O}_3$ , imparts a definite resistance to mildew; 1 per cent. of chrome checks mildew growth. Even in the presence of alkali (it had earlier been shown that alkali favours mildew growth in wool) the restrictive properties of the chrome were apparent.

#### Action of Chrome

Examination of the behaviour of commercially dyed wool when inoculated with mould spores also indicated that chrome has an important retarding influence on mildew growth.

Now the action of the chrome may depend on various factors. Firstly, the chrome may render the wool substance less soluble and thus impoverish it, considered as a source of food for mould fungi. Such micro-organisms can assimilate only soluble substances. Thus the chrome may render the wool substance less accessible. This appears to be a likely explanation, for the following solubilities obtained by boiling dyed wools with distilled water and estimating the nitrogen content of the aqueous extracts certainly show that chromed wool is the less soluble:—

| Treatment of wool and dyestuff.                 | Nitrogen in wool extract.<br>(Mg. per 100 g. of wool). |
|---|--|
| A.—Dyed with Eriochrome Black T (chromed) ..... | 27.23  |
| B.— ditto (not chromed) .....                   | 36.18  |
| C.—As A. without dyestuff .....                 | 39.81  |
| As C. without dyestuff and chrome .....         | 41.48  |
| Dyed with Alizarin (chromed) .....              | 29.43  |
| Dyed with fast acid dye (not chromed) .....     | 49.58  |

The susceptibility of wool to attack by mildew is increased by an increase of humidity, so that if the chrome affected the moisture absorbent properties of the wool it could thus influence its resistance to mildew attack. A series of experiments showed that chromed wool was just as moisture absorbent as ordinary wool; the protective influence of the chrome is thus not due to rendering the wool less moist.

The amount of chrome normally present in a wool which has been dyed and chromed is much less than the 0.5 per cent. found necessary to retard mildew growth. Thus the mere presence of chrome in the dyed wool is not the reason for the action of the chrome. Summarising his investigations Burgess concludes that the protective influence of chrome in dyed wool is due to the impoverishment of the wool as a source of nutrient such as is required for supporting the life of mould fungi.

In reviewing the destructive action of light and micro-organisms on dyed textile materials it is evident that, although the dyestuffs themselves may exert a protective or harmful influence there is also the important fact that the processes of dyeing and the assistants or mordants employed are able to confer a resistance to deterioration. In order to combat these destructive influences satisfactorily it is necessary to have available colourless dyestuffs of an inert character which can be applied to the textile material by simple means (but not be readily removed by washing treatments); these substances should have properties such that they render the energy of light ineffective or the life of micro-organisms impossible. No doubt research will eventually result in their discovery.

#### German Coal Tar Production

With local demand and supply of coal tar about equalised, summer production in the German coal tar market was from 15 to 20 per cent. less than capacity. Germany produces some 1,500,000 metric tons of coal tar annually, of which 1,200,000 tons are consumed by distillation plants. The market for road-building tar picked up after a rainy summer. Briquet pitch enjoyed a considerable import. It was quoted from 45 to 48 marks per ton. Tar oils were selling well, partly due to protective tariffs encouraging their use as fuel and motor oils. They sold from 80 to 90 marks per ton. The naphthalene and anthracene market was quiet. Crude naphthalene was quoted at from 50 to 60 marks per 100 kilos and anthracene residues at 35 to 40 marks per ton.—U.S. Trade Commissioner William T. Daugherty, Berlin.

## Basic Intermediates for Dyestuffs : No. XXXIX.—Derivatives of the Anthraquinone Series. Part 2.—The Indanthrenes

By "Consultant"

WHEN a brand name is used subsequently for the systematic chemical name of a substance or group of substances, there is nearly always some confusion. This is undoubtedly true of the so-called "Indanthrene" series of colours. Originally, the term "Indanthrene" was used by the Badische Anilin-und Soda-Fabrik company as the trade name for a series of vat dyes, which might or might not be derived chemically from the substance that we now term Indanthrene. The series corresponded closely with the Duranthrene and Chloranthrene series of the British dyes, of a decade ago, and a number of these dyes are still used commercially, and constitute, therefore, a problem of interest in regard to their intermediates.

The old Indanthrene trade group was derived from five distinct types of substances :—

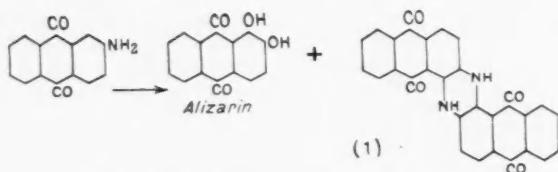
1. The Indanthrene derivatives proper.
2. The Violanthrone derivatives.
3. The Pyranthrone derivatives.
4. The anthraquinacridone series.
5. A miscellaneous group of dyes of unknown constitution.

### The True Indanthrenes

Indanthrene itself (1) is the parent intermediate for the preparation of the dyestuffs of this class, although it may be used as a dyestuff itself. The members of this class, past and present, are :—

Indanthrene (Caledon Blue, Duranthrene Blue RG).  
Dibromindanthrene } Indanthrene Blue CE and GC.  
Tribromindanthrene }  
Dichloroindanthrene. Indanthrene Blue GCD.

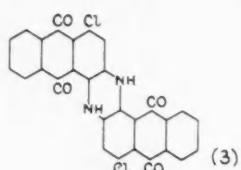
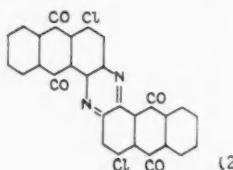
Indanthrene itself was first prepared by the caustic fusion of 2-aminoanthraquinone at 200–300° C., a process which was first introduced by Bohn. At the present time it is usual to add potassium nitrate to the melt in order to destroy other compounds which are produced as by-products. Apparently alizarin is one of the earliest substances formed during the course of this melt, and the production of a certain amount of this substance is inevitable. Its presence does not interfere with the isolation of the indanthrene. If the fusion is conducted in the absence of an oxidising agent the Indanthrene obtained is not a homogeneous product and consists of two products, which, for want of better terms have been differentiated as Indanthrene A and Indanthrene B, the former being the more valuable from the dyestuffs point of view. Indanthrene B has little affinity for vegetable fibres and manufacturing operations are carried out with the aim of eliminating the B compound as far as possible.



The caustic fusion for Indanthrene is conducted along orthodox lines. The caustic alkali (1 cwt.) is melted in a suitable kettle and mixed with potassium nitrate (5 lbs.) a small quantity of which is sufficient. The temperature is maintained at 200° C. and the finely ground amino anthraquinone is added with stirring. The temperature of the melt should be raised slowly to 250° C. and maintained at that point for half an hour. The cooled melt does not contain the Indanthrene as such, but in the form of a reduced compound which probably contains two phenolic hydroxyl groups. When air is passed through an aqueous solution of the cooled melt the indanthrene separates in dark blue flocks. It contains a little indanthrene B, which can be separated by converting the dye to its dihydro derivative by hydrosulphite reduction, and liberating the colour from the purified sodium salt of the dihydro body, by aeration.

### Halogenated Indanthrenes

The dichloro derivative of indanthrene is obtained by means of an intermediate, the identity of which has not been completely elucidated. The process used affords almost the only application of the mixture known as "aqua regia" as a chlorinating agent. The process is as follows :—

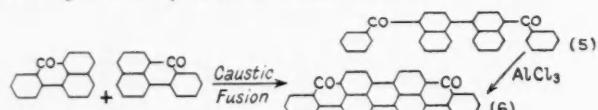


The so-called fuming nitric acid of a density 1.5 is mixed with two and a half times its weight of commercial concentrated hydrochloric acid (density 1.17). To this mixture is added, slowly and with stirring, one-sixth of its weight of very finely powdered indanthrene, which is kept at 10° C. during the addition. After the addition has been completed the temperature of the mixture is raised to 50° C. and maintained at that point for three hours. The process of chlorination involves as an accessory process, oxidation and the compound which separates on pouring into water is a chlorinated azine, with a constitution represented by (2). When this compound is dissolved, as for vat dyeing, in a solution of alkaline hydro-sulphite, reduction of the azine group occurs, and the actual dichloroindanthrene may be obtained by the aeration of such a reduced solution.

The dibromoderivative, on the other hand, may be obtained by direct bromination, the solvent employed being concentrated sulphuric acid. The indanthrene is usually brominated in about 15 lb. charges. Each charge is dissolved in the strong acid (2½ cwt.) and the solution warmed to 70° C. The bromine (9 lbs.) is slowly added, but the reaction takes a considerable time, and it is best to keep the solution warmed to 70° C. for 24 hours. The compound is actually isolated by pouring onto ice, filtering the product and washing with dilute alkali, followed with pure water. It is used as Indanthrene Blue GC. Both of these compounds have an exceptional fastness to chlorine.

### Violanthrones

The preparation of violanthrone (6) on a large scale is effected by the caustic fusion of benzanthrone (4) two molecules of which unite with the elimination of four atoms of hydrogen. Its constitution is shown by the formation from 4:4'-dibenzoyl-1:1'-dinaphthyl (5), which loses six hydrogen atoms on heating with anhydrous aluminium chloride.



The caustic fusion of benzanthrone is an operation which proceeds on straightforward lines, and is commenced at about 180° C. The actual formation of the violanthrone takes place when the temperature is raised to 240° C. and the isolation of the dye is effected by a simple boiling with hot water. The following are among the so-called "Indanthrene" dyes, which are in reality derived from the violanthrone nucleus :—

1. *Violanthrone*. Indanthrene Dark Blue BO, Caledon Purple.
2. *Chloroviolanthrone*. Indanthrene Violet RT.
3. *Isoviolanthrone*. Indanthrene Violet R. extra. Caledon Brilliant Purple R.
4. *Dichloroisoviolanthrone*. Indanthrene Violet 2R.
5. *Dibromoisoviolanthrone*. Indanthrene Violet B. extra.
6. *Aminoviolanthrone*. Indanthrene Green B.

Of the above compounds the preparation of violanthrone

has already been dealt with. The formation of the chlorovianthrone is best accomplished by grinding the vianthrone to a very fine paste with nitrobenzene and suspending the paste in nitrobenzene. Chlorination takes place when gaseous chlorine is passed through this suspension and the end of the reaction is judged by withdrawing a sample, adding alcohol filtering and reducing the precipitate with hydrosulphite. The

solution so obtained should have a blue-green colour. The chlorovianthrone is isolated by pouring into alcohol with subsequent filtration.

The isovianthrone compounds are produced from isovianthrone which is, in turn, prepared from chlorobenzanthrone, a process which has been described in detail in No. XXVIII of this series.

## Dyes and Their Application: Recent Technical Progress

By L. J. Hooley

### Stable Diazo Preparations

REFERENCE has already been made in previous articles dealing with azo derivatives in the SUPPLEMENT to the production of stable diazo preparations. These save the dyer the trouble of treating the amine with sodium nitrite and acid before coupling, and eliminate disadvantages such as the presence of nitrous fumes in the dyehouse. The previously described products include nitrosamines, zinc chloride double salts with diazonium compounds, and preparations of these with alum, aluminium sulphate and other salts.

### Diazonium Fluorsulphonates

During the last year or so a number of new methods dealing with the same compounds have been patented. Several of these make use of fluorine compounds; the simplest of these are the diazonium fluorosulphonates— $N : N.FSO_3$ . A simple diazonium salt, such as the chloride or sulphate, is treated with free fluorosulphonic acid, when an almost theoretical yield is obtained. As an example, 28.3 parts of 4-chloro-2-aminotoluene are dissolved in 60 parts of hydrochloric acid and diazotised with 14 parts of sodium nitrite. A slight excess of 40 per cent. fluorosulphonic acid is then added and the mixture cooled, when the required product comes down in pure crystals and can be filtered off and dried, preferably in vacuum. It is readily soluble in water and stable, and can be used in dyeing or as an insecticide. Fluoro-sulphonic acid is, of course, unlike chlorosulphonic acid which cannot be used in the presence of water (B.P. 303,527, I.G. Farbenindustrie A.-G.).

### Increasing the Solubility of Diazonium Fluoborate

The diazonium fluoroborates which were described in G.P. 281,055 and *Ber. d. dentsch. Chem. Ges.*, **60**, 115 and 1,186, are not always sufficiently soluble for practical dyeing purposes, especially with ice colours, in printing, and in dyeing with standing baths. Much more concentrated solutions can, however, be obtained if the fluoborate is dissolved with a soluble neutral salt of a metal which has greater affinity for the fluoroboric acid residue than for the acid residue with which it is initially combined. Salts of groups I and III, including ammonium salts, are the most suitable. As an instance, *p*-nitrophenyldiazonium fluoborate may be warmed with potassium chloride solution and a solution about 60 per cent. stronger obtained. The potassium fluoborate formed in the double decomposition separated out may be filtered off. An insoluble salt is not always formed, this depends upon the reactants (B.P. 332,030, I.G. Farbenindustrie A.-G.).

### Use of Complex Fluoric Acids

More complex fluoric acids, such as titanium hexafluoric, aluminium hexafluoric, tin-hexafluoric, antimony tetra- and hexafluoric and zinc-tetrafluoric, have also been brought into service. The free acids react with diazonium salts, giving crystalline salts of surprising stability. They are unaffected by rubbing and on heating do not explode but only decompose. They can be recrystallised from hot water and burn with difficulty or not at all.

### *o*-Aminophenol Ether Derivatives

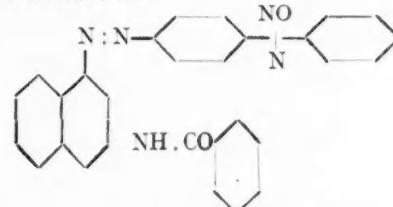
Certain monazo compounds obtained from *p*-aminoazo compounds can be used in solid form without first converting them to derivatives of the types which have already been mentioned. These are the *p*-aminoazo bodies which contain an alkoxy-, aralkoxy- or aryloxy-group in ortho position to the amino group, and after conversion to their diazo compounds in the usual manner they are precipitated from solution by adding an alkali halide, a sulphate, sulphuric acid, a heavy metal salt or an arylsulphonate. Precipitation is not always necessary and then the diazo compound is mixed with one of the substances mentioned before drying. As an example, the *p*-aminoazo body, 4-nitrobenzeneazo-4'-amino-3'-methoxy-6'-

methylbenzene is diazotised in dilute hydrochloric acid, and common salt added, when the diazonium chloride, separated in crystalline form, is filtered and dried at about 40-50° C. (B.P. 305,498, I.G. Farbenindustrie A.-G.)



*Azo-nitroso-Diphenylamines*

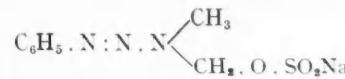
Comparatively recently, blue dyes of the Naphthol AS series have been obtained by coupling a 2:3-hydroxy-naphthoic acid arylide with a diazotised aminodiphenylamine. These aminodiphenylamines do not, however, couple very well, but the corresponding N-nitroso bodies are satisfactory. These are easily obtained by using an excess of nitrous acid in the diazotisation, when a second molecule reacts with the NH group. After coupling on the fibre the dyestuffs have the general constitution.



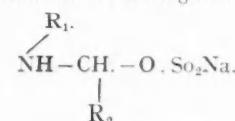
The nitroso group gives a shade of a reddish-brown character, but it is easily removed by treating on the fibre, and the desired blue obtained. So that the dyer shall not have to make an exception with these colours and go back to carrying out his own diazotisation, it is desirable that these nitroso-diazo salts shall also be available in convenient form. Unfortunately, it has proved impossible to prepare these in the ordinary dry form on a technical scale because the nitroso group splits off during the drying. However, the difficulty can be overcome by mixing the aminodiphenylamine with a dry alkali nitrite and a suitable solid substance to liberate nitrous acid from the nitrite. These products then give the nitrosodiazos compound on dissolving in water or acidified water. Dry 4-methoxy-4-diazoniumbenzene sulphonate is, for example, mixed with dehydrated sodium sulphate, dry sodium nitrite and sodium bisulphite. (B.P. 307,890, I.G. Farbenindustrie A.-G.)

### Aminomethyl Sulphurous Acid Derivative

Another device for protecting a diazo group is condensation with an aminomethylsulphurous acid to give a compound such as



(cf. Wallach, Ann. 235,233). They are soluble in water and with acid split again into their components, they are also stable in the dry form. Acids of the general formula,



where R<sub>1</sub> is hydrogen, an aliphatic or hydroaromatic radicle or the residue of an alkylsulphurous acid, and R<sub>2</sub> is hydrogen or an alkyl, aryl or aralkyl residue. The only body of this

type previously known was imino-dimethyl-sulphurous acid, obtained by the action of ammonia on two molecules of formaldehyde sulphurous acid. The new bodies can be prepared from ammonia or primary aliphatic or hydroaromatic amines on bisulphite compounds of aldehydes (B.P. 309,610, I.G. Farbenindustrie A.-G.). The condensation products can be used as plant protecting media as well as in dyeing and printing.

**Decomposition in Presence of Iron**

An allied process, which strictly speaking does not come into the same class as those above, can be described here as it deals

with the use of diazo solutions in dyeing. Diazo solutions, as is known, may decompose in the presence of iron, and as iron vessels often cannot be avoided, this is a nuisance, especially in working with standing baths. According to B.P. 326,866 (I.G. Farbenindustrie A.-G.) a substance which will oxidise ferrous iron to ferric will reduce this decomposition and nitrous acid is found to be a suitable substance for this purpose, in spite of the fact that it has been found to have a deleterious action on diazo compounds themselves. A table is given showing the comparative rates of decomposition with and without nitrous acid.

## **Dyestuffs Markets: The Month's Business in Review**

**From Our Own Correspondents**

### **Lancashire**

As previously anticipated in this column, the Dyestuffs (Import Regulation) Act is to lapse at the end of the period of ten years originally fixed. The effect upon the British dyestuff-making industry will be, according to some of the principal speakers for the industry, a very serious one, and may lead eventually to a considerable reduction in the numbers of trained organic chemists employed in the dye-making industry, with evil consequences to chemical industry in general in the country. The attitude of the majority of dye-users on the question appears to be in favour of the termination of the restrictions imposed upon the import of foreign dyestuffs, whilst at the same time they are in full agreement with the thesis that any diminution in dye-making activities in this country, especially in regard to the employment of trained chemists, may well be serious to the country.

So far as the textile industries of Lancashire are concerned, the British dye-makers have been successful in the last few years in establishing a high level of confidence, based upon consistency and good quality of the colouring matters produced, and there would appear to be every reason to suppose that British dye-makers will receive the very loyal support of the Lancashire textile industries. There is a feeling that with the inclusion of the British Dyestuffs Corporation, Ltd., in Imperial Chemical Industries, Ltd., on the one hand, and the close connection existing between the Clayton Aniline Co., Ltd., and the Swiss Group (and, through the latter, the German I.G.) on the other hand, the danger of squeezing and the ultimate elimination of the British dye-making industry by foreign competition is improbable in the extreme. It is felt that if any policy of price cutting or dumping is indulged in by foreign manufacturers, the I.C.I., for example, is sufficiently powerful not only to meet this competition, but to retaliate.

The dyer and printer is not, of course, concerned mainly with obtaining his dyestuffs at the lowest possible price. It is, however, of importance to him that he should not be called upon to pay more than the dyer and printer on the Continent or elsewhere, and, further, that he should have immediate access to the latest products capable of producing improved or novel effects, whatever their origin. Few users can be found to support the contention that in such qualities as purity of shade, fastness, and consistency British dyes are in any way inferior to those produced in Switzerland and Germany. So far as the writer's experience goes, dye-users in Lancashire appreciate the efforts which have been made by British manufacturers during the past ten years, and are prepared to continue their support. They are relieved, however, by the knowledge that they will have access to German or other products which are not made in this country, without any of the inconveniences which may arise from the need for import licences.

### **Scotland**

BUSINESS has been fairly good in Scotland on the whole during the past month, considering the present circumstances. Of course, the Dyestuff Act is ousting all other subjects as a topic of interest. The loss of the amendment in the House of Commons last week was another disappointment, but as the House of Lords will also deal with the matter, hope is not lost, especially as it appears that the tide of feeling is running more strongly every day in favour of continuance. As a result of the uncertainty, many users have expressed themselves even more

definitely in favour of the makers. A case was heard the other day of a colour user who told a traveller that in the past he had used a certain amount of German colour in addition to British, and that he had the new German price list in his hands for next year; but if the Act in fact did come off, he had made up his mind to support the British industry as far as possible by buying nothing but home colours.

### **Dyestuff Traders and the Act**

#### **Decision on Key Industry Duty**

In a circular to members, the British Chemical and Dyestuffs Traders' Association states that at the time the Government notified their decision to allow the Dyestuffs Act, 1920, to lapse on January 14 next, inquiry was made as to what would be the position as regards liability to duty of those products marked "D" in the list of articles chargeable with Key Industry duty. The Board of Trade have now advised the Association:—

"Regarding the position on the expiry of the Dyestuffs Act of the products marked with the prefix 'D' in the list of articles chargeable with key industry duty, they understand that the position will remain as heretofore, excepting that no licence will be required for the importation of these materials."

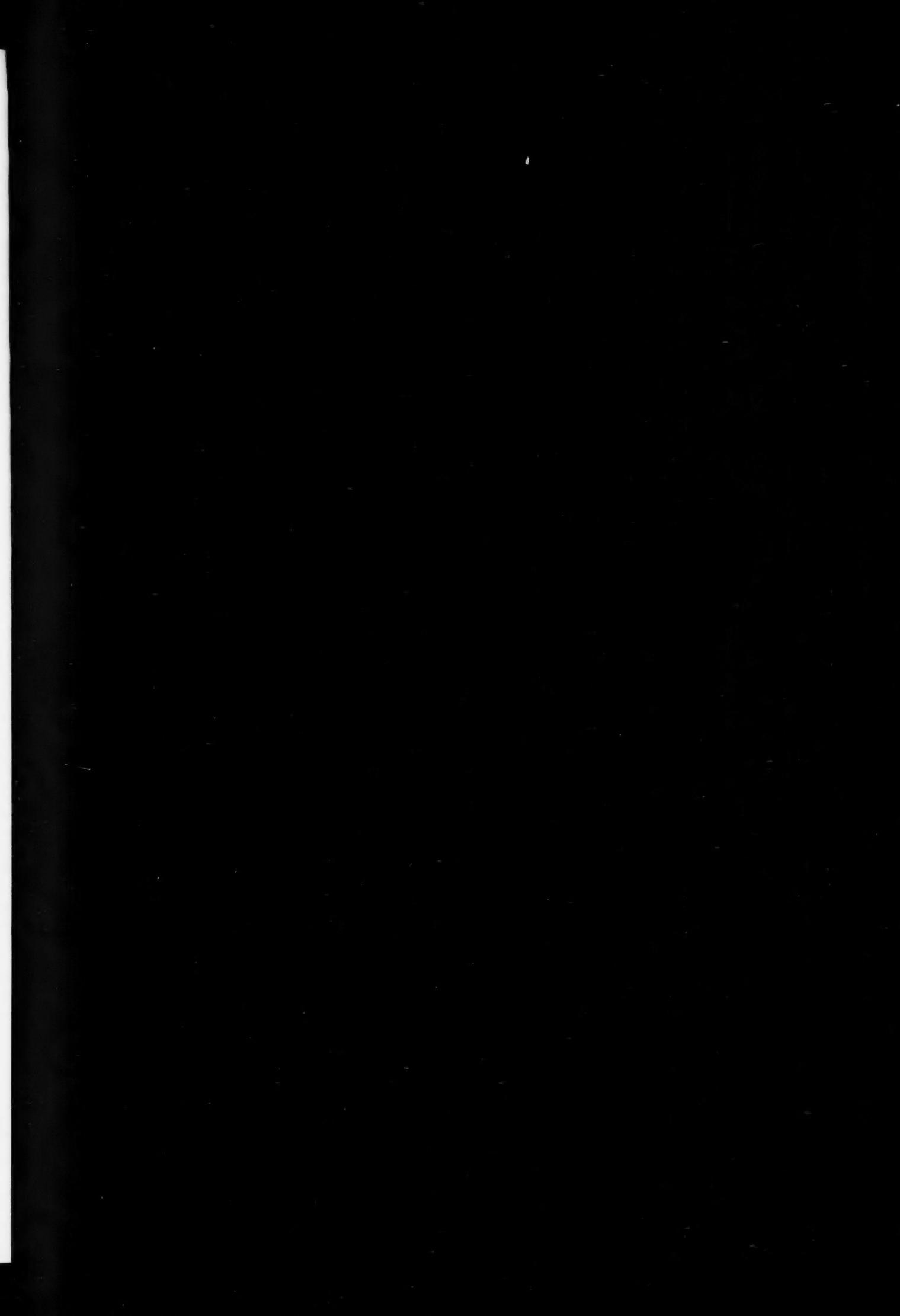
Although specified in the Key Industry List, the products marked "D" are exempted from duty under certain conditions. The schedule to the Act provides for duty on synthetic organic chemicals (other than synthetic organic dyestuffs, colours and colouring matters imported for use as such) and organic intermediate products imported for their manufacture. Therefore all products mentioned in the Key Industry List of dutiable articles prefixed "D" will not be charged with key industry duty. These are: (1) Synthetic organic dyestuffs, colours and colouring matters *imported for use as such*; and (2) organic intermediate products imported for use in the manufacture of synthetic organic dyestuffs, colours and colouring matters.

If the products falling under (1) or (2) are imported for use in any industry *other than* that of dyestuffs, colours or colouring matters, then duty will be charged on such imports. The Association has asked Customs what evidence they will require at the time such imports are cleared, and declared to come under (1) or (2). Their reply will be circulated in due course.

It will be noted that the question of liability or otherwise to key industry duty is to be decided not on the article itself, but on its ultimate use.

#### **F.B.I. Plea for Non-Party Inquiry**

A plea for a non-party inquiry into the Government's decision to allow the Dyestuffs Act to lapse was made on Wednesday at a meeting of the executive committee of the Federation of British Industries. The following resolution was carried unanimously: "The executive committee of the Federation of British Industries is strongly of opinion that in a matter of such importance from the point of view of national prosperity and security, there should have been an impartial inquiry into all the facts of the case before the decision was taken to allow the Dyestuffs (Import Regulation) Act, 1920, to lapse in January, and urges on the Government the necessity of extending the Act for such period as may be required to enable a judicial investigation on non-party lines to be made."





# Metallurgical Section

Published in the first issue of "The Chemical Age" each month

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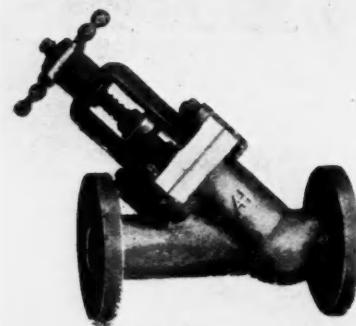
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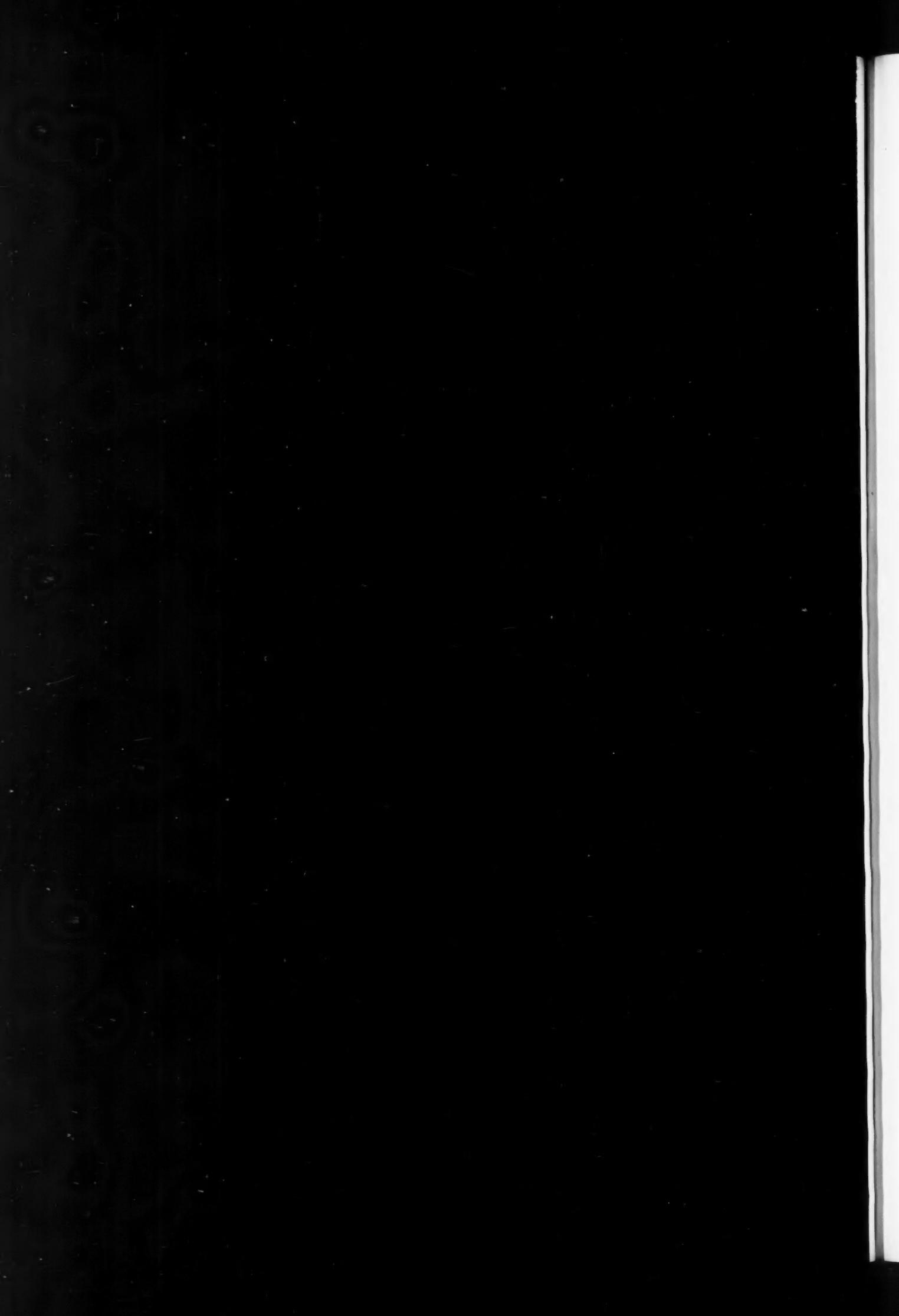
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This summarised account of physical chemistry has been written for candidates who are reading chemistry up to the final stage of their university course, and are conversant with elementary mathematical methods. It may also be used with advantage by those who are taking open scholarships at Oxford and Cambridge, and by masters giving advanced courses in public and secondary schools. The whole ground is covered, and care has been taken to include all fully established recent developments, without minimising the importance of the older foundations on which the science rests.

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# Monthly Metallurgical Section

Published in the first issue of "The Chemical Age" each month.

**NOTICE.**—Communications relating to editorial matter for our Monthly Metallurgical Section should be addressed to the Editor, THE CHEMICAL AGE, Bowyer House, 154, Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.

## The Effect of the Rate of Cooling on Steel

By J. M. Robertson, D.Sc., Ph.D., A.R.T.C.

Accepted theories of the effects of heat-treatment on steel are critically examined by Dr. Robertson in Paper No. 59, published by the Safety in Mines Research Board on "The Effect of the Rate of Cooling on the Structure and Constitution of Steel" (H.M. Stationery Office, 2s. net). Some of Dr. Robertson's conclusions are published below.

Of the numerous experimental methods that have been used in investigating the phenomena of heat-treatment, thermal analysis and microscopic examination are undoubtedly the most important. As far as carbon steels are concerned, the method of thermal analysis has been most effectively applied to investigation of the changes that take place during cooling at normal rates, whereas the effects of more rapid cooling have been chiefly studied by means of the microscope. Assuming that steel could be regarded in the same way as rock, the first step in interpreting its microstructure was the identification of the components or micro-constituents. The micro-constituents identified in steel were called ferrite, cementite, pearlite, sorbite, troostite, martensite and austenite, and it was considered that whatever the heat-treatment and composition of a piece of steel, its microstructure could always be described in terms of the combination and arrangement of this set of micro-constituents.

### The Original Theory

In the first explanation of the occurrence of sorbite, troostite and martensite, it was supposed that these constituents corresponded with a series of intermediate states through which the steel passed during the normal decomposition of austenite. This view of the occurrence of these constituents is given in most of the standard works, and according to it, increasing the rate of cooling is simply a means of causing the temperature to fall more rapidly than the transformation can proceed. The theory, which for convenience may be referred to as the original theory, accounts for the effect of the rate of cooling in the following way. When cooling is very rapid, the change from austenite to pearlite passes through one stage only, *i.e.*, from austenite to martensite—the first step in the complete transition. With a slower rate of cooling the change proceeds to a greater extent, *i.e.*, it passes through martensite to the next state, troostite. When the rate of cooling is only a little faster than the normal rate, the steel passes through martensite and troostite to sorbite, the last intermediate state before pearlite.

The original theory of the relations of martensite, troostite and sorbite to each other and to the normal iron-carbon diagram, was at one time widely accepted. But many alterations have from time to time been proposed and various modifications of the theory are now current. These theories are all based on the original theory or on one or other of the two principal modifications, and, although there are many possible combinations of the distinct parts of the original theory and the modifications subsequently proposed, all the fundamental conceptions are included when the original theory and the two principal modifications are described.

The modification of the original theory is the result of the application of methods of thermal analysis to rapid cooling, and it has now been demonstrated by means of cooling curves, magnetic curves and dilatation curves of carbon and alloy steels, that increasing the rate of cooling lowers the temperature at which the decomposition of austenite takes place. This fact has been demonstrated so frequently, by so many workers and in so many different countries, that it is now almost universally accepted, and the recognition of this relation between the rate of cooling and the temperature of the transformation constitutes one modification of the original theory.

As a result of investigations, the following connection

between the rate of cooling and the temperature of the transition has been established:—As the rate of cooling is progressively increased, the temperature at which the transformation begins is gradually lowered. When a certain rate of cooling is attained, however, there is a sudden discontinuous lowering of the change to the neighbourhood of 300° C. That rate of cooling required to produce this sudden lowering of the change is known as the "critical rate of cooling." Rates of cooling considerably less than this critical rate, produce slight lowering of the transformation temperature; when the rate of cooling approaches the critical rate the transformation takes place partly at a high temperature and partly at a low temperature; and when the rate of cooling equals or exceeds the critical rate, the transformation takes place entirely at a low temperature. The point at which the normal transition from austenite to pearlite takes place during cooling is known as the  $Ar_1$  and on the suggestion of Portevin the change that takes place when the  $Ar_1$  is slightly lowered is now known as the  $Ar'$ , and the low temperature change as the  $Ar''$ .

### General Conclusions

The general conclusions from the investigations on which the present paper is based are that the effect of variation in the rate of cooling cannot be adequately accounted for by supposing that accelerating the cooling is simply a means of producing in turn the three constituents, sorbite, troostite and martensite. The microstructure of steel may be varied almost without limit by varying the composition, the time and temperature of heating, and the rate of cooling. If all the complicated variations in structure and constitution that may be produced have to be described in terms of a limited number of distinct constituents, then these constituents cannot be exactly defined, for, within the meaning of each of the terms martensite, troostite and sorbite, many different states must be included.

In its wider sense the nomenclature associated with the original theory of heat-treatment has an undoubted usefulness. It enables an approximate idea of the observed structure to be expressed, and in this way it serves to distinguish between the structures of properly quenched, improperly quenched, air cooled and slowly cooled steels. The structures obtained by these treatments do not, however, supply complete information about the effect of the rate of cooling on steel; consequently a complete theory of heat-treatment cannot be based on the examination of specimens cooled under these conditions. To obtain a complete record of the structures formed by cooling steel at progressively increasing rates, it is necessary to cool the steel at progressively increasing rates. That is what has been done in the present investigation, part of which is described in this paper.

### Four Series of Structures

By different methods of cooling, four series of structures may be obtained. The first series is produced by cooling at various rates through the  $Ar'$  range; this series includes all the structures consisting of ferrite and pearlite and of pearlite alone. The second series of structures is obtained by continuous rapid cooling which suppresses the  $Ar'$  and causes the  $Ar''$  to take place at low temperature. This series of structures includes those structures that are recognised as characteristic of quenched steels.

The third series of structures is that obtained by cooling in media held at different temperatures. In this way the  $Ar'$  may be suppressed, and the rate of cooling then retarded so that the  $Ar''$  change may take place at constant temperature. The fourth series of structures is similar to the third, except that the steel is withdrawn from the cooling medium before the change is complete. The change is completed during cooling to ordinary temperature, and the resulting structure consists of two parts, that formed at the temperature of the medium, and that formed during subsequent cooling.

The constitution of the product of the decomposition of austenite depends on the temperature of decomposition, and on the rate of abstraction of heat while the decomposition is in progress. When the change begins at the normal temperature during slow cooling, the austenite changes to an aggregate of almost pure  $\alpha$ -iron and cementite. When the change begins at a low temperature during rapid cooling, the austenite changes to a solid solution of carbon in  $\alpha$ -iron. Any state intermediate between these extremes may be obtained by suitably controlling the cooling, that is, the carbon may be divided in any proportion between the  $\alpha$ -solid solution and cementite.

The structure of the product of the decomposition of austenite depends on the constitution, and on the way in which the constitutional changes take place. Classified according to the manner of their formation, the structures in steel may be divided into two main groups—(1) structures whose form is determined by the habit of one constituent, and (2) structures whose form is determined by the fact that two constituents separate concurrently.

The general arrangement of all ferrite-pearlite structures is determined by the manner in which ferrite develops from austenite. The structure of the grains of pearlite is, however, determined by the simultaneous growth of ferrite and cementite. In all steels containing more than 0.28 per cent. of carbon, certain structures consisting entirely of simultaneously formed ferrite and cementite may be produced; these have been called the eutectoid structures.

The  $Ar''$  change is led by the formation of  $\alpha$ -solid solution. The structures formed in the manner of this change are therefore related to the ferrite structures, of which they are a continuation. In steels containing less than 0.15 per cent. of carbon, the transition from the ferrite structures to the  $Ar''$  structures is gradual, but in steel containing more carbon, there is a break in the structural continuity due to the formation of eutectoid structures. It is the difficulty of suppressing the formation of these eutectoid structures that is responsible for the large temperature interval between  $Ar'$  and  $Ar''$  during continuous cooling. In steels containing less than 0.15 per cent. of carbon, this discontinuous lowering of the change is not so well marked.

Any structure formed during cooling may be altered by re-heating to temperatures below the lower limit of the normal critical range. This alteration does not take place in such a way as to produce any other structure formed during cooling. The changes that take place during re-heating are comparatively simple and depend on two factors only: the separation of carbon from solution and the change in shape of the cementite formations. If the change during cooling has taken place at the  $Ar'$  point with the production of a ferrite-cementite aggregate, the only change that can take place on reheating is a change in the shape of the cementite plates, which are slowly converted to globules at temperatures just below the normal critical range. Simultaneously with the globularisation of the cementite, aggregation or coalescence of the ferrite plates takes place.

All aggregate structures are affected by reheating in the manner described above, and the various forms of the eutectoid structure, including the fan structure, as well as the aggregate structures formed by interrupting the cooling between  $Ar'$  and  $Ar''$ , temper in such a way that the original plates of cementite are converted to globules. In all cases the original form of the structure may be recognised because of the orientation of the ferrite matrix and the distribution of the cementite globules.

The only structures that temper in a different manner from those described above, are the structures that contain considerable quantities of  $\alpha$ -solid solution, for during reheating, decomposition of this  $\alpha$ -solid solution takes place. This decomposition does not proceed, as is commonly believed,

by the sudden precipitation of all the carbon in the form of infinitely small particles and the subsequent coalescence of these particles to form larger and larger globules. Decomposition actually takes place by the gradual separation of carbon as the temperature is raised. During the early stages of tempering, the cementite formations cannot be seen; they may either be plates or globules. In any case, the  $\alpha$ -solid solution becomes an aggregate, which, when the tempering temperature is further raised, tempers as described above, that is, by globularisation and growth of cementite particles, and coalescence of the ferrite (or  $\alpha$ -solid solution).

### Changes in Metal Prices

#### Cost and Control Factors

In the course of his presidential address at the annual general meeting of the Institution of Mining and Metallurgy last week, Mr. James G. Lawn, A.R.S.M., said that variations in the prices of minerals and metals over short periods of years touched the mining engineer most closely, and brought in its train difficulties and hardships innumerable. When the engineer was called upon to advise as to the opening up of a deposit he might have very clear ideas as to methods of mining, metallurgical treatment, and cost of production, but the question of the price to be received for the product was one of grave difficulty and yet might be the determining factor. If prices fell too severely there might be only the alternatives of running at a loss in the hope of better prices, or of closing down the mine. The circumstances connected with the occurrence and production of minerals and metals were such that in certain instances control of prices had been established, and in other instances great efforts were being made to secure that end. The danger of control leading to excessive prices was largely provided against by the fact that such prices would limit demand, cause substitutes to be used, and stimulate competitive production.

#### Problem of Control

Considering the question of control in relation to the two fundamental factors of supply and demand, it was clear that control could only be applied to supply. Demand could not be controlled, though stabilised prices tended to make demand more uniform. It depended on various economical and psychological factors which were often obscure. The supply of minerals and metals could in some cases be controlled because of the limited number and extent of known deposits which could be worked profitably, in other cases because of metallurgical difficulties in extracting a marketable product. In the former category would fall diamonds, nickel, cobalt, potash, and nitrate; in the latter, aluminium and magnesium.

The four base metals, copper, tin, lead and zinc, were so widely distributed and were worked by such numerous owners in many countries that any general agreement by the producers of a particular metal was exceedingly difficult to secure. Even when the bulk of the owners had agreed to control output, the small number who remained outside could be exceedingly troublesome when demand slackens.

Up to the present, perhaps, the most successful of the associations was in connection with copper, where, by amalgamations and purchasing of controlling interests, a considerable proportion of the production of the world had come into few hands and a strong organisation existed for the control of production and the regulation of the price of electrolytic copper. In the case of tin, a producers' association had recently been formed, and restriction of output started. A policy of research and propaganda has also been laid down. Lead would apparently be more easy to control than either copper or tin, because supplies were not capable of indefinite expansion, and world stocks were comparatively small, but so far little had been attempted in that direction.

It was not long since a world shortage of lead was looked upon as probable, but the discovery in 1925 of the differential flotation process, whereby lead and zinc ores in intimate association could be effectively separated, altered the position substantially; though in spite of this fact lead may be the first of the base metals to fall short in supply.

Zinc was, perhaps, in the weakest position of the four metals. All attempts at control by cartels had hitherto failed. No doubt, however, on the basis of the experience gained, and by serious effort on the part of those interested, much could be accomplished.

## The Invention and Characteristics of Silumin

By W. von Eichorn

An account of the invention, characteristics and technical development of Silumin, by W. von Eichorn, appears in the "Periodic Review" of the Metallgesellschaft A.G., Frankfort-on-Main, which since 1920 has carried out extensive experiments in the production of this alloy.

IN September, 1920, the Metallbank und Metallurgische Gesellschaft A.-G. received the offer of an invention which seemed to indicate the possibility of making a very considerable step forward in the technique of aluminium casting. Test experiments carried out in the metallurgical laboratory of the Metallgesellschaft confirmed the statements of the inventor, and it was possible to start experimental operations on a factory scale in January, 1921.

The circumstances which led to the invention of silumin are of no little interest, and we therefore give the following brief sketch of its history. The inventor, Dr. Pacz, a Hungarian by birth, before emigrating to America, had studied chemistry at Charlottenburg and was then for some time employed as a chemical expert with the Auer-Gesellschaft. Soon after he had settled in the United States, Dr. Pacz, in pursuance of his work with the Auer-Gesellschaft, resumed his experiments with among other things the tungsten lamp (Halbwatt-Lampe), the filament of which is encased in an atmosphere of nitrogen or argon. The problem which specially interested him was, how to produce a filament with a micro-crystalline structure and so avoid the expansion and deflection to which the tungsten filament was liable under intensive heating, with resultant diminution of its illuminating power.

The solution of this problem led him further to the idea of making similar experiments with the compound of aluminium and silicon, known for its very coarse crystalline structure. This alloy attracted his interest because he knew that the presence of silicon in an alloy generally produces a thin running casting, and increases the resistance to acids, for example, in cast-iron.

Following similar methods to those he had used with the tungsten lamp, he succeeded in producing a eutectic alloy of aluminium and silicon of a surprisingly fine structure and in preventing the formation of the coarse silicon crystals usually met with in such alloys.

This "graining process" was, and still remains, the basis of the patents applied for by Dr. Pacz and further developed by the Metallgesellschaft. Dr. Pacz had already recognised that this fine grain is brought about in the first place by the presence of sodium, whether added in metallic form or as a compound. Further observations showed that sudden and uniform cooling, such as, for instance, occurs in casting in chilled mould of good conductivity, produces a similar fineness of grain. This problem of reducing the size of grain has in the course of time attracted the interest of all the leading metallurgists. Although explanations and solutions have been suggested from many quarters, it is still a subject of dispute. An early conjecture of Cochralski's, who assumed that undercooling effects were involved in the phenomenon, has much in its favour.

In strong contrast to the doubts which existed as to how the phenomenon was to be explained, the physical fact itself was from the first obvious. Observation by the naked eye alone of the fracture of a properly produced eutectic alloy of aluminium and silicon at once opened up fascinating prospects in regard to its properties. Instead of the usual crumbly surface presented by the fracture of an aluminium-zinc alloy, for instance, or an aluminium-copper alloy in which coarse crystals are embedded, the aluminium-silicon alloy showed a steel-like, silky, lustrous fracture in which no microscopic crystals were visible. The originality and importance of Pacz's discovery, which is rightly looked upon as an important piece of pioneer work, became still more evident when fractures of normal and special castings were compared. The fracture of a normal casting shows crystals with facets up to 1.0 sq. mm. in size lying close together in irregular order, appearing either very dark in colour or glittering like rock crystals according to the way in which the light is reflected. A mere glance at such a conglomeration of coarse crystals sufficed to explain why aluminium-silicon alloys had been hitherto considered unsuitable for technical purposes.

The industrial experiments gave results which were in

gratifying agreement with the statements of the inventor and with further investigations meanwhile undertaken in the metallurgical laboratory of the Metallgesellschaft. When the melt was correctly treated, the structure, even of large pieces, always showed the characteristic fine fracture. The tests for hardness and elasticity gave high average values and maximum values such as were hitherto unknown for aluminium castings.

All this indicated clearly the technological importance of the new alloy. It was soon proved that the alloy possessed further valuable qualities, some of which were already known from the literature on aluminium, while others were new. Especially notable were its excellent casting qualities, which far surpassed all hitherto known limits in regard to the size of aluminium casts, the small shrinkage, the low coefficient of thermal expansion, the high thermal conductivity, relatively high electrical conductivity, and an excellent resistance to corrosion.

The good casting qualities and the high degree of resistance to attack by chemicals have been throughout the highest value in the further development of the alloy and have perhaps contributed more to its popularity than its outstanding mechanical properties. The other qualities mentioned above, however, have all materially helped to promote its utilisation for various special industrial purposes.

The new alloy "Silumin" was not intended to compete with aluminium and such of its alloys as had already been utilised with success as building materials, rather was it meant to be a means of shedding economic fetters, increasing working capacity and avoiding waste of material. As soon, therefore, as practical work had gone far enough for a general estimate of the qualities of silumin to be formed, a research programme was drawn up in accordance with these principles with the support of various other interested undertakings and operations were commenced.

This programme embraced railway requirements, such as coke trucks, undercarriages for goods trucks, roofs, doors, windows, journals and other fittings for cars, brake sets, pumps, lubricators, journals, slippers and other fittings for locomotives, motor car parts, and in the chemical industry, substitutes for building materials which are quickly worn out by chemical action, various massive apparatus which require frequent dismantling and re-erection, the costs of which are out of proportion to the material value, e.g., large boilers and vats, evaporation tanks, drying apparatus, settling tanks, crystallising pans, similar apparatus used in the fermentation industry and in the manufacture of soap and perfumery.

It may be that some of the items of the initial programme appeared to be over bold to many critics at the time, and possibly may still excite a sceptical smile. Creation and criticism stand at all times side by side and each has its right of existence.

In eight years Silumin has been adopted in almost all branches of industry in a thousand different forms, from the small die casting weighing only a few grams to heavy sand castings of up to 1,500 kg. Over and above this, the development of this one type of aluminium alloy has given a powerful and helpful impetus to the development of all light metals and their utilisation in industry.

### Metallurgical Works in Canada

THE Acting High Commissioner for Canada in London has received from the Mines Branch of the Dominion Department of Mines at Ottawa a revision of List No. 1-1, giving a directory of "Metallurgical Works in Canada," including iron blast furnaces; steel furnaces; nickel, copper, silver, lead and zinc smelters and refineries; and other metallurgical works. Copies of the list will be available shortly for distribution, and application should be made either to the Mines Branch at Ottawa, or to the Secretary, Office of the High Commissioner for Canada, The Canadian Building, Trafalgar Square, London, S.W.1.

## Metallurgical Topics: Monthly Notes and Comments

From Our Own Correspondents

### National Physical Laboratory

PROBABLY by collusion with the Clerk of the Weather, the sun usually shines on the occasion of the annual inspection, by the General Board, of the National Physical Laboratory at Teddington. Friday of last week was no exception to the rule, and the inspection was made, with the assistance of an unusually large attendance of visitors, in delightful weather. The laboratory continues to grow, and why it does so is made plain in the report for the year 1929, recently issued. The range and diversity of the research work grows with the increasing growth of industry. Nowhere is this astonishingly accelerated pace more clearly demonstrated than in the Departments of Metallurgy and of Engineering, under the respective superintendence of Dr. Rosenhain and Sir Thomas Stanton. Many of the exhibits and demonstrations on view in these and other departments served to show how fine a line separates what used to be called "pure" research from investigations of what are described as being of a "practical nature," a distinction which at the National Physical Laboratory may, for all practical purposes, be regarded as obsolete. The two categories dovetail into, and are indeed complementary to, each other. Thus "creep" investigations are being carried out simultaneously both in the Engineering and in the Metallurgy Departments. Their objects are, however, essentially different.

### Creep Tests at the Laboratory

THE importance of creep in these days of intensive pressures and super-temperatures in engineering is incontestable. The experiments in the Engineering Department, on metals and alloys at high temperatures, have hitherto been confined to iron and steel, as used for turbine blading, at temperatures between 450° C. and 650° C. They have since been extended to 700° C. In addition to creep determinations, the short-time tensile properties and notched bar impact strength of a 0.51 per cent. carbon steel, and a 3 per cent. nickel steel, both heat treated, have been carried out. In comparison with a normalised 0.51 per cent. carbon steel, both the heat-treated carbon steel and the nickel steel had slightly high limiting creep stresses at 300° C., but were somewhat inferior at 500° C. The energy to fracture in impact for temperatures up to 600° C. was higher for the nickel steel than for the carbon steels, and the heat treated carbon steel was superior to the normalised carbon steel.

### Behaviour of Alloy Steels at High Temperatures

A 0.24 carbon steel containing 1.7 per cent. of manganese investigated between the temperature range of 300° C. to 600° C. was found to have a longer life for high stresses than a normalised 0.24 per cent. carbon steel, but, on the other hand, its superiority in this respect decreases with decrease in stress, while both steels have limiting creep stresses of the same order. These, and various creep rates and limiting creep stresses for stainless steels, and various alloy steels, serve to show how these metals behave, and may be expected to yield methods for routine testing, and comparative results. The investigations in the Metallurgy Department have, however, a different object, and show not so much how metals will behave as the structural and other changes they undergo when subjected to stresses at high temperatures. They include the mode of deformation, flow, and fracture, and give useful indications of the methods to be adopted—or avoided—in manufacture, and although, therefore, they are of the nature, in the first instance, of pure research, they have immediately practical and useful implications. Another series of experiments is being conducted to discover metals capable of resisting prolonged loading at high temperatures when exposed to the influence of chemical attack as well.

### Sound Aluminium Alloy Castings

THE production of sound metal, in the sense of the elimination of blowholes, is, of course, one of the main preoccupations of the metallurgist. Some very interesting work has been done in Dr. Rosenhain's department, in this direction. In the rolling mill house were specimens of pure aluminium, and of Y alloy and 12 per cent. silicon alloys which had been

subjected to a special and ingenious treatment, by which the dissolved gases, which cause porosity in castings, can be removed and at the same time a molten alloy made to solidify in castings with a fine grained texture. The method consists in bubbling through the molten metal, at slight pressure (sufficient to enable adequate circulation to be effected), titanium tetrachloride, which subsequently, at the temperatures involved, volatilises and escapes. The effect produced is probably more mechanical than chemical, but in the result the influence of the process on the refinement of the grain and on the elimination of blowholes and porosity is truly remarkable. In this department, too, considerable progress has been made in the production of pressure die castings in aluminium alloys. This investigation has been carried out on behalf of the British Non-Ferrous Metals Research Association and has as its object the determination of the tensile properties of such alloys when forced into a mould under pressure, about 90 lb. per square inch being the pressure available in the machine devised for the purpose. Considerable difficulty was experienced in the satisfactory filling of the mould, as there is a tendency for the metal first entering it to solidify as a complete shell, thus entrapping air as a cavity in the finished casting. Improved design of the moulds and more appropriate placing of the gates have largely minimised this defect, while providing the mould with a kind of overflow chamber has solved it almost entirely. The excess metal entering this chamber can be made to carry with it the entrapped air, thus producing a cavity outside the test piece proper. Surprisingly high tensile results have been obtained with pressure-cast test pieces, particularly in the case of heat-treated "Y" alloy, although in this material considerable irregularities are observed.

### I.B.F. Convention at Middlesbrough

MIDDLESBROUGH was this year selected at the meeting place for the annual convention of the Institute of British Foundrymen, which continues to make marked progress and deals both with ferrous metal founding and occasionally with non-ferrous metal practice. The programme of papers, however, dealt in the main with cast iron and steel subjects, two of the most interesting dealing with the remarkable divergencies between the properties of certain mixtures for foundry work as compared with what might be expected in view of the actual chemical composition of the mixtures employed, and particularly of the actual pig irons used in making these mixtures. This problem is one which has long exercised the minds of foundry men, and has become more acute of late, owing to the increasing tendency to use more and more steel in mixtures corresponding with what is now termed "Semi-steel." The paper on "The Economic Utilisation of British Pig Iron Resources" skirts round this subject and is valuable chiefly for the very extensive list of analyses of typical pig irons available for foundry purposes, but, as was pointed out in the subsequent discussion, too much reliance is often placed on the uniformity of pig iron composition as judged from the standpoint of what it ought to be, or is supposed to be, taking makers' analyses as typical and fully representative of the run of pig they purport to relate to, as compared with what, over a large batch, it actually is. On the other hand, the paper by Messrs. Ridsdale failed to point out the reasons for these discrepancies, which have recently been dealt with in an interesting manner by Mr. Fletcher, the well-known consultant to the British Non-Ferrous Metals Research Association, who assigns reasonable causes for the variations so often found between theory and practice. At the Convention the only non-ferrous paper this year was one by Mr. F. W. Rowe, on the Founding of Bronze Gear Blanks, which dealt, in the author's well-known and efficient style, with problems involved in this important branch of foundry work.

### American Investigations on Manganese Distribution in Steel

It is impossible to rate too highly the value of the technical researches of all kinds, results of which are published from time to time by such American institutions as the Bureau of Mines, Washington, the University of Illinois, and the

University of Michigan, or to avoid some expression of regret that our own admirable institutions of a similar nature do not publish their own results in the same way so as to render them available at moderate cost to all those interested therein. Usually such work as is done in corresponding laboratories in Great Britain, if published at all, is communicated only to certain societies and institutes, and thus becomes available only to members. Such abstracts as appear in the technical Press are rarely of sufficient length to supplement the necessity of going to the original source—an expensive matter if it involves membership of the publishing institution. The American way is the better. Thus, the Bureau of Standard Monographs are available to all.

One of the latest, by B. M. Larsen, deals with the effect of manganese on the distribution of carbon in steel. It is found by the author that three factors essentially determine the modifying effects of manganese on carbides in the iron-carbon system. They are (1) its tendency to extend the temperature range of stability of gamma iron or austenite; (2) its formation of a carbide more stable than cementite, the corresponding carbide of iron; and (3) the fact that manganese atoms can hardly diffuse at all through the iron space lattice at ordinary heat-treating temperatures at which carbon atoms diffuse quite freely.

#### Extension of the Eutectoid Range

In the case of steels containing more than one per cent. of manganese dendritic segregation occurs in cast metals, and in rolled or forged bars induces a fibrous pattern not affected by ordinary heat treatment until from 1,100° to 1,200° C. is reached, when the manganese atoms gain some slight freedom of movement in the iron space lattice. Heating at 1,300° C. enables the manganese to diffuse uniformly and removes the dendritic segregation. Dendritic patterns are also caused in other circumstances by phosphorus and by arsenic, but the presence of these elements raises the  $Ar_3$  point of pure iron and the ferrite areas formed correspond to the dendritic fillings. Above the  $Ar_3$  point the carbon atoms probably diffuse uniformly in the gamma iron lattice, irrespective of the non-uniform distribution of the manganese atoms. The author finds some evidence favouring the conclusion that, with an increasing percentage of manganese, what might be called manganese-cementite tends to form, containing perhaps an increasing proportion of molecules of  $Fe_2MnC$ . Some interesting indications likewise emerge relative to the widening of the eutectic point in pure iron-carbon alloys round about 0.90 per cent. of carbon, when the manganese percentage increases: with 3 per cent. of manganese the eutectoid range extends from 0.60 to 0.92 per cent. of carbon, owing probably to the very small temperature interval between  $Ar_3$  and  $Ar_1$  in the given range of carbon content.

#### A New Work on Aluminium

THE present price of copper has several important economic implications, one of which is that there is less inducement to use aluminium for electrical cables and other purposes for which it has hitherto largely superseded copper. It would appear, however, that the light metal and its alloys still attract more interest from a metallurgical point of view than all the other "base metals" put together, as its literature enormously exceeds, both in quantity and quality, that of copper, tin, lead, or zinc. The latest addition thereto is the large and authoritative work in two volumes just published by the McGraw-Hill Book Co., in their "Chemical Engineering Series" (*The Aluminium Industry*). The two volumes aggregate over 1,250 pages, and the first volume deals with "Aluminium and its Production," while Vol. II is entitled "Aluminium Products and their Fabrication." They are by J. D. Edwards, F. C. Frary, and the distinguished metallurgist Zay Jeffries, names which may be taken as guarantees of the thoroughness and accuracy with which this great work is written.

Hitherto Dr. Anderson's excellent book has held the field, and while it still remains one of the best books on the subject, the new work is far larger, and of course brings it more up to date. The first volume begins with the story of aluminium, a fascinating chapter in the history of human discovery, and then, as its title indicates, deals with the aluminium industry generally, noting its development in the principal countries, the occurrence and treatment of its ores, and the processes

by which the metal is extracted and the plant and equipment employed for the purpose. In the second volume the commercial history of aluminium is told by Mr. S. K. Colby; J. D. Edwards deals with the physical and chemical properties of the metal and its working, and a number of other contributors, including R. S. Archer and E. H. Dix, deal with the constitution and properties of aluminium alloys, melting, casting and heat treatment; coating and finishing; the uses of aluminium, and, indeed, every aspect of the manufacture of aluminium and aluminium alloy products. The work is certainly the most comprehensive hitherto published, and a very notable contribution to the literature of an exceedingly important industry, while its scientific value is all that the names of the distinguished joint writers would lead the reader to expect.

#### Research on Copper

RESEARCH on impurities in copper for the British Non-Ferrous Metals Research Association has been continued with particular assiduity during the past year at the National Physical Laboratory, states the annual report of Dr. W. Rosenhain, Superintendent of the Department of Metallurgy and Metallurgical Chemistry. The effects of bismuth, arsenic, antimony and oxygen upon copper have previously been determined so far as these elements occurring separately are concerned. In refinery or "tough pitch" copper, however, a number of these elements are generally found to be present simultaneously in various proportions, and it is therefore one of the objects of the investigation to determine the influence which combinations of these elements exert upon the more important properties of the material. Certain combinations of these metals, two at a time—viz., bismuth and arsenic, arsenic and antimony, and to some extent bismuth and antimony, in the absence of all but very small quantities of oxygen, have been studied, while some progress has also been made in the study of the effects of these elements singly and together in the presence of rather larger amounts of oxygen. A few selected alloys containing oxygen, bismuth and arsenic have been prepared and tested, while work on the oxygen-bismuth-antimony series has also progressed sufficiently to give some indications of the effects of oxygen in conjunction with bismuth and antimony together. The complete elucidation of the somewhat complex relationships of this group of impurities is obviously a matter requiring extensive investigation, but the results already obtained are of very great interest and importance, because they serve to show that while some of the views formerly held about the relative importance of these impurities are fully justified, others do not appear to be so well founded.

#### "Poling" Copper

ONE of the most interesting consequences of the investigation is the light which it throws upon the well-known process of "poling" copper, thereby bringing it into the condition known as "tough pitch." It has been known for some time that in copper of extreme purity, where such substances as arsenic, antimony and bismuth are absent, the complete removal of oxygen is attended with beneficial results. In copper of lower general purity, however, it is found that if the removal of oxygen is carried too far, the material becomes "over poled" and loses its satisfactory properties. These facts are explained by the results of the present investigation, which serve to show the manner in which the presence of a suitable proportion of oxygen assists in preventing or removing the evil effects which certain of the impurities tend to exert upon the copper in the absence of oxygen. The interaction of the different impurities, however, is of such a nature that the exact proportion of oxygen required will vary according to the relative amounts of the impurities present. The condition of "tough pitch" is reached when, in the natural course of the poling operation, the amount of oxygen has adjusted itself to this proportion.

While the impurities above mentioned are perhaps of primary importance, as regards their effect on the properties of furnace-refined copper, the presence of nickel in this material may also prove to be of some importance particularly if it is found, as is suspected, that nickel affects the behaviour and influence of the other impurities. It is hoped that the study of the effect of nickel, together with some of the other impurities above mentioned, may be undertaken in the near future.

**Some Inventions of the Month**  
By Our Patents Correspondent

Abstracts of other Patents of metallurgical interest will be found in our Patent Literature, published weekly in THE CHEMICAL AGE.

**Alloys**

A NICKEL-IRON alloy powder for use in the manufacture of cores of electromagnetic apparatus is obtained by heating a mixture of the oxides of the metals in a reducing gas. The iron oxide used is other than that produced by heating iron carbonyl. One or more additional oxides—e.g., cobalt oxide—may also be present. See Specification 327,865, dated April 18, 1929, by General Electric Co., Ltd., of London, and S. V. Williams and G. R. Polgreen, of Wembley, Middlesex.

**Lead Alloys**

METHODS of increasing the resistance of lead to sulphuric acid are described in (1) Specification 326,818 (Imray), and (2) Specification 327,106 (Imray), of a Patent of Addition thereto, both being communications from I.G. Farbenindustrie Akt.-Ges. of Frankfort-on-Main, Germany, dated December 18, 1928. According to (1) the lead is alloyed with 0.01—0.05 per cent. of nickel, with or without an addition of not more than 0.1 per cent. of selenium. Reference is made in the Provisional Specification to the addition also of copper, silver and tellurium. According to (2) the lead is alloyed with not more than 0.1 per cent. of selenium, without the addition of nickel.

**Metal Carbonyls**

ACCORDING to Specification 327,956 (Johnson), a communication from I.G. Farbenindustrie Akt.-Ges., of Frankfort-on-Main, Germany, dated December 14, 1928, metal carbonyls are obtained by causing reducible metal compounds—e.g., sulphides, carbonates or oxides—to interact with carbon monoxide at high temperatures and pressures. Temperatures of 200-400° C. and pressures of 20-1,200 atmospheres are specified. The reduction may be facilitated by the presence of copper, lime or caustic alkalis. Decomposition of the carbon monoxide may be suppressed by the addition to it of a sulphur compound—e.g., carbon disulphide—or by mixing alkali or alkaline earth sulphides with the metal compound.

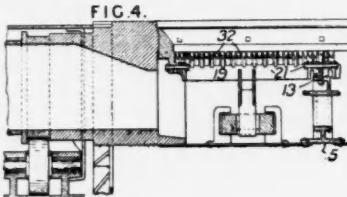
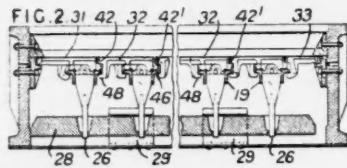
**Non-Porous Metal Articles**

A PROCESS for obtaining non-porous metal articles is described in the Specification 326,536 (Johnson), dated December 13, 1928, a communication from I.G. Farbenindustrie Akt.-Ges. of Frankfort-on-Main, Germany, of a Patent of Addition to Specification 311,141. According to the parent Specification porous articles substantially composed of metals, and in particular electrodes suitable for use in storage batteries with alkaline electrolytes, are made by subjecting a metal in a finely divided condition obtained by thermal decomposition of the corresponding metal carbonyl to a heat treatment and/or a pressure treatment without melting, whereby the finely divided metal may be moulded into any desired shape. According to the present invention, non-porous articles are made similarly by the employment of higher temperatures or higher pressures or of more prolonged treatment or of repeated treatments. A decarbonizing treatment, e.g., with hydrogen, may be effected simultaneously with the heat and/or pressure treatment to produce, for example, iron foil for magnetic purposes.

**Sintering Ores**

GRATES for use in ore-sintering pans described and illustrated in Specifications 326,819 and 326,834, dated December 19, 1928, are so constructed that on tilting the pan for dumping, positive movements are imparted to the grate-bars in order to dislodge by a shearing action any material that may have become fused to the bars or lodged in the openings. According to Specification 326,819, by J. E. Greenawalt of New York, U.S.A., the grate is provided with bar-carrying frames so arranged that the tilting of the pan serves to rock the frames to move some bars in one direction and others in a different direction. The frames 19 are carried by pins 21 journalled in brackets 13 carried by beams 5, which stiffen the pan transversely. The end grate-bars 31, 33 rest in recesses in the pan walls, and the intervening bars 32 are each provided at one end with lugs 42, 48, and at the other end with a lug 42<sup>1</sup>. Each lug 42<sup>1</sup> and a flange 46 of the adjacent frame 19 are located between the lugs 42, 48 of the next bar in the line. The bars in the next line are oppositely arranged so that

adjacent ends of bars are engaged by opposite flanges of a frame 19. Upon tilting the pan the frames are rocked by the engagement of an arm 26 on each frame with a slotted bar



326,819

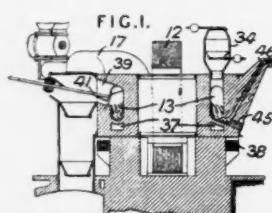
28 sliding in guides 29, and as one flange 46 of each frame is raised and the other lowered alternate lines of bars are raised and lowered. According to Specification 326,834, by T. M. Alexovitis of Bayonne, New Jersey, and J. E. Greenawalt of New York, U.S.A., the arrangement is similar, but certain of the bars are twice as long as the others, long and short bars being in alternate rows. The short bars are held by flanges to rock with the frames, while the long bars are each held between two frames so that they receive no tilting movement, but are moved upwards.

**Raising Fatigue Strength of Metals**

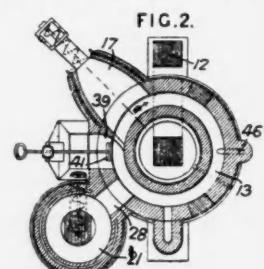
SPECIFICATION 327,660 (Mond), a communication from I.G. Farbenindustrie Akt.-Ges., of Frankfort-on-Main, Germany, dated August 15, 1929, describes a method of increasing the fatigue strength of a metal or alloy by subjecting it to continuous dynamic stress which is raised as the fatigue strength rises, but is always kept below the actual fatigue limit. The invention is described with particular reference to alloys of magnesium and aluminium for use in aircraft engines.

**Reducing Ores**

A PROCESS for reducing iron ores in an electric furnace is described in Specification 327,687, dated January 4, 1929, by J. Herrmann, of Magdeburg, and B. G. Franzen, of Berlin, Germany, and L. M. Hubbard and E. R. Zacharias, of New



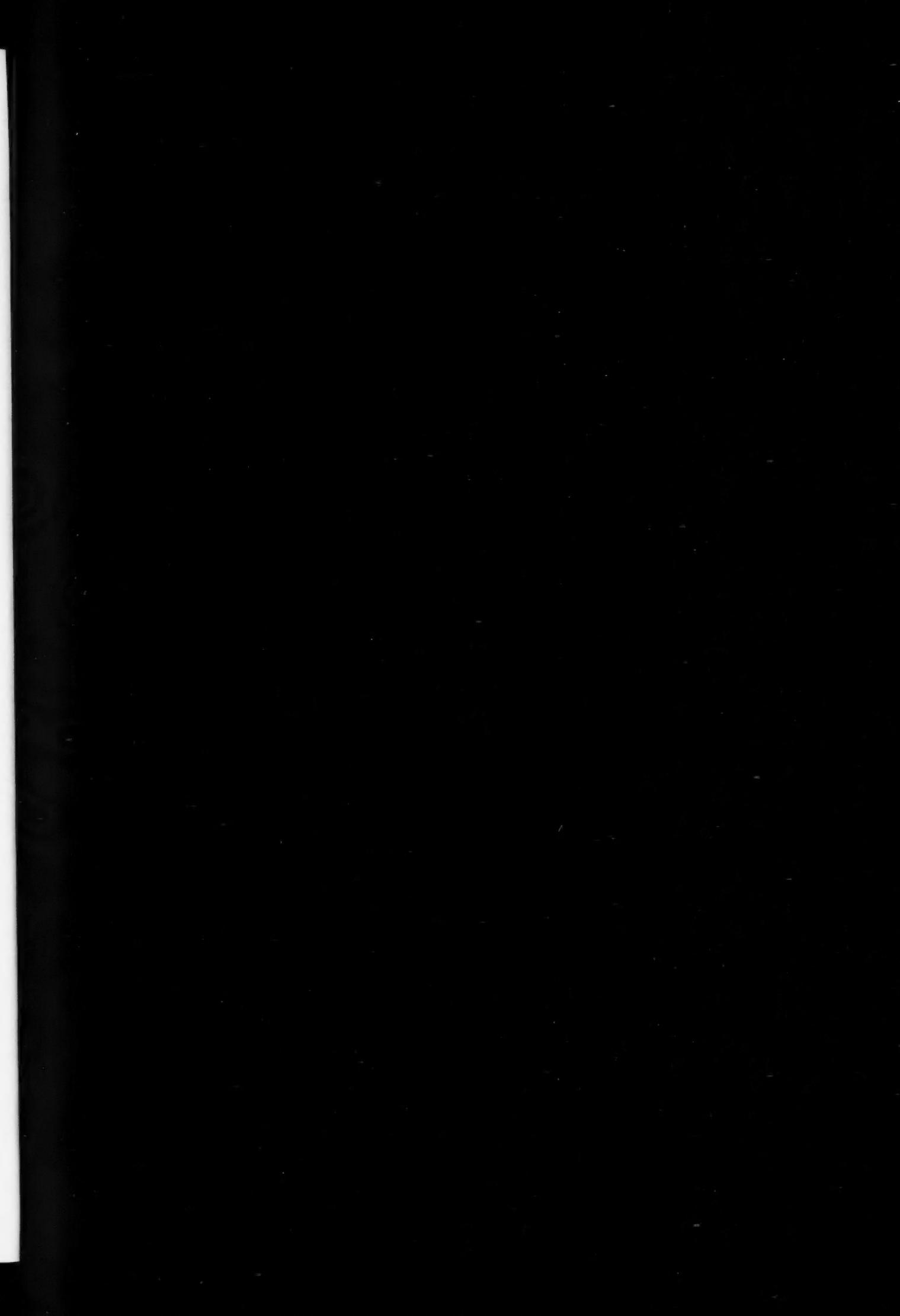
327,687

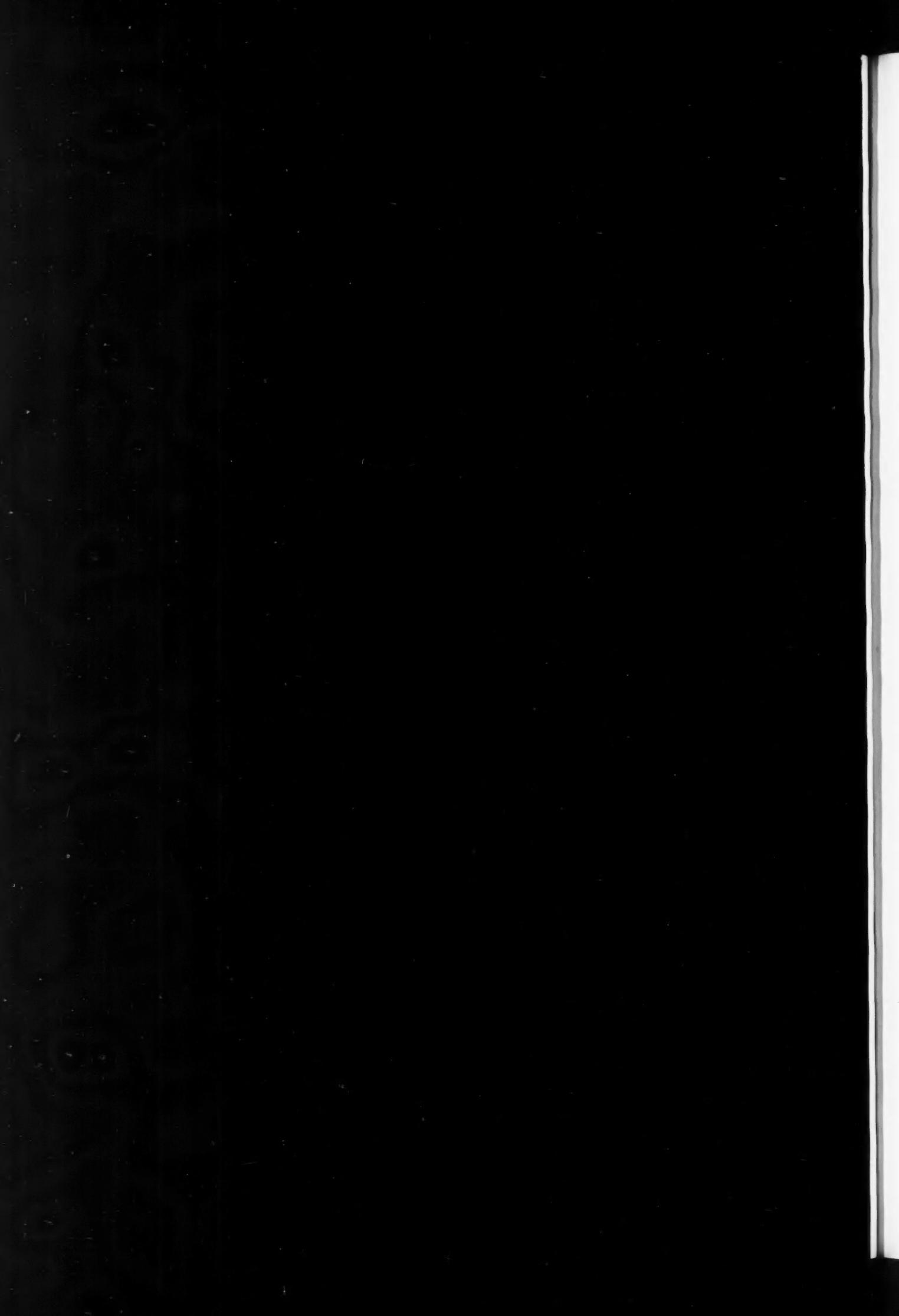


York, U.S.A. The ores melted in an electrically heated chamber 17 pass into an annular chamber 13 where they are subjected to the action of hot combustion gases under pressure supplied from a gas producer 21. The chamber is provided with electrical heating devices 38 and heating flues 37. An electromagnet 12 draws the particles of iron or steel toward it for removal through a gate 45 or a passage 46. Flux or additional ore may be supplied from a hopper 34. The slag and other impurities are removed by a rake 41 through an opening 39.

**Refining Metals**

In a process for refining metals and alloys, oxides and carbon are added to the molten bath in the proportions required to form carbon monoxide. The process is particularly applicable to iron and copper and alloys thereof. See Specification 327,570, dated April 5, 1929, by W. Reitmeister, of Havel, Germany.





## Monthly Metallurgical Section

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**NOTICE.**—Communications relating to editorial matter for our Monthly Metallurgical Section should be addressed to the Editor, THE CHEMICAL AGE, Bouvier House, 154, Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.

### Cadmium versus Zinc Plating as Rust Preventive

By Dr. W. S. Patterson

Some interesting tests on the relative values of zinc and cadmium electro deposits in resisting corrosion under varying conditions were described by Dr. W. S. Patterson in the course of a recent paper before the Electroplaters' and Depositors' Technical Society. Dr. W. H. J. Vernon presided, and an animated discussion followed the reading of the paper.

INTRODUCING his paper, which bore the title of "The Resistance of Electrodeposits to Corrosion, with Special Reference to Cadmium and Zinc," Dr. Patterson said there were three distinct aspects of this problem. There was (1) corrosion of the metal constituting the coating, assuming it was a continuous envelope sealing completely the metal it is protecting; (2) corrosion where the protective coating is not a complete enveloping surface; and (3) that due to inherent weakness to corrosion or breakdown, internal to the deposit itself, for example, stresses in deposits, traces of bath liquor remaining in the deposit, or of pickling solution in the pores of the base metal, etc.

Very frequently in practice coatings fail rather because of the causes enumerated under headings (2) and (3) than because of the total corrosion of an initially sound coating. In point of fact, Dr. Patterson said that in his experience (2) appears to be the greatest cause of failure, and after that (3). Very seldom do high-grade electrodeposited coatings, such as are covered by (1), break down in actual service unless the coating used was totally unsuited for the type of exposure to be employed.

The deposits used by the author were prepared from solutions of the following compositions:—

|                          | Zinc Solution. | Cadmium Solution.     |
|--------------------------|----------------|-----------------------|
| Zinc sulphate .....      | 300 gms.       | Cadmium Oxide 24 gms. |
| Sodium chloride .....    | 13 "           | Sodium cyanide.. 66 " |
| Boric acid .....         | 20 "           | Water ..... 1 litre   |
| Aluminium sulphate ..... | 26 "           |                       |
| Dextrose .....           | 13 "           |                       |
| Water .....              | 1 litre        |                       |

The thickness of the coatings used was 0.65 mil. in the case of cadmium, and 0.70 mil. in the case of zinc. The base metal used throughout was mild steel containing 0.18 per cent. carbon. Before plating, the specimens were pickled in dilute sulphuric acid, scoured with pumice, rinsed in dilute potash, washed in tap water, and finally were boiled in distilled water and dried. They were then weighed and placed in the baths for the requisite time. The deposits obtained in both cases were clean, close grained, and showed no signs of pinholing.

#### Indoor Corrosion Tests

For indoor corrosion tests plates were mounted in a room away from chemical pollution, the average humidity of which was 55 per cent. during the period of exposure. Before being exposed the plates were rubbed with 00 emery paper and then weighed. After 90 and 250 days' exposure the plates were removed, brushed with a soft camel hair brush and re-weighed. The following table gives the mean results with the two sets of plates.

|              | Weight Increments, Milligrams.<br>90 days. | 250 days. |
|--------------|--|-----------|
| Zinc .....   | 1.1  | 2.7       |
| Cadmium..... | 1.8  | 2.0       |

The corresponding graph shows that the zinc plates corroded in such a way that the curve is practically a straight line. The corrosion products are of such a character that once produced they do not influence the course of the subsequent corrosion. With cadmium, however, the curve indicates a gradual retardation of the attack and the underlying metal is protected. The corrosion products formed on cadmium

during the indoor corrosion therefore protects the metal, and resulting from this, cadmium would appear to be the more suitable coating for such conditions of exposure.

It is necessary, however, to remember that these coatings are generally used to protect iron. Clearly then, either coating will be quite an effective protection to the iron provided it is properly applied, and either is likely to be very durable so far as corrosion is concerned. If the claims made for cadmium can be justified that it can be more easily deposited and that the bath is more consistent in its output, it would appear that for the indoor type of exposure examined an appreciable argument could be put forward for the choice of cadmium.

#### Saturated and Polluted Indoor Atmosphere

Specimens similar to those used in the preceding experiments were exposed in the atmosphere of a large swimming bath. The relative humidity was high and dew point conditions were frequently established. This was particularly so at times during the winter period (January-March), when the bath was used only for part of the week and the temperature was very considerably reduced when not in use. Further, the atmosphere contained traces of chlorine. This condition of exposure is, of course, very severe, but is no more so than might occur in certain types of industrial practice. The mean results from these plates of each type of plating are given in the subjoined table.

| Days.   | Zinc Plated.                     |                      | Cadmium Plated.                  |                       |
|---------|----------------------------------|----------------------|----------------------------------|-----------------------|
|         | Weight increment,<br>Mill. grms. | Mean<br>grms./sq.in. | Weight increment,<br>Mill. grms. | Mean<br>grms./sq. in. |
| 96 ...  | 6.1<br>6.5<br>6.5                | 7.1<br>7.9<br>6.7    | 5.2                              | 5.9                   |
| 175 ... | 10.0<br>9.9<br>9.5               | 13.4<br>14.1<br>12.9 | 8.1                              | 1.1                   |
| 272 ... | 18.8<br>18.8<br>18.5             | 34.3<br>34.1<br>33.6 | 1.5                              | 2.8                   |

These results were somewhat unexpected, as numerous statements concerning the relative merits of the two coatings are certainly in favour of cadmium. The atmosphere concerned is undoubtedly a highly corrosive one, but nevertheless the test is equally severe for both types of coating. Upon the completion of the tests a careful examination of the specimens revealed no signs of rusting. The corrosion phenomenon described is that produced on a perfectly sound deposit.

#### Exposure of Specimens Outdoors

Simultaneously with the foregoing experiments an examination of the corrosion of similar plates exposed outdoors in the Clerkenwell area of London was investigated. Exposure of this type differs from the preceding types in that the specimens are at irregular intervals subjected to washing or erosion by rain. The results are given in the next Table.

It is apparent that the destruction of the coating occurs very much more rapidly in the case of cadmium than in the case of zinc. Once the coating is destroyed there appears to be appreciable evidence to show that the breakdown with

cadmium is very rapid, but at the time of writing this has not yet occurred.

| Days.   | Outdoor Exposure (June to March). |                        |                         |                        |
|---------|-----------------------------------|------------------------|-------------------------|------------------------|
|         | Zinc Plated.                      |                        | Cadmium Plated.         |                        |
|         | Loss in weight, gms.              | Mean Loss, mg./sq. in. | Loss in weight, gms.    | Mean Loss, mg./sq. in. |
| 93 ...  | ·0299<br>·0266<br>·0306           | ·0290<br>2·4           | ·1153<br>·0988<br>·1359 | ·1166<br>9·7           |
| 185 ... | ·1770<br>·1822<br>·1759           | ·1783<br>14·8          | ·5238<br>·5288<br>·5308 | ·5278<br>43·9          |
| 167 ... | ·2198<br>·2271<br>·2009           | ·2359<br>19·6          | ·8863<br>·8724<br>·8611 | ·8733<br>72·7          |

In view of the severity of the attack of the cadmium-plated specimens it was decided to expose plates of pure cadmium and pure zinc along with further electroplated specimens. These were exposed in the months of January, February and March for 76 days. The results are given in the following table, and these figures confirm the previous results.

| Exposure Outdoors (January–March) 76 days. |               |              |                 |
|--|---------------|--------------|-----------------|
| Loss in Weight, mg./sq. in.                |               |              |                 |
| Pure Zinc.                                 | Pure Cadmium. | Zinc Plated. | Cadmium Plated. |
| 6·5  | 17·7          | 8·2          | 21·4            |

It is not without significance that the zinc-plated specimens which had already been exposed since the previous June only lost 4·8 milligrams per sq. in., whilst the new plates lost 8·2 milligrams per sq. in. in a slightly shorter period. In the same period the new cadmium plates lost 21·4 milligrams per sq. in., whilst those which had been exposed since the previous June lost 28·8 milligrams per sq. in.

#### Mechanism of Atmospheric Corrosion

In the indoor unsaturated corrosion test the coating formed upon zinc is discontinuous, and oxidation occurs at a uniform rate by diffusion of oxygen through the porous scale. In the case of cadmium the coating formed is protective, and is of such a character that it retards the attack.

With the indoor saturated and polluted atmosphere test the composition of the corrosion products plays an important part. In view of the high humidity it is likely that the action is essentially electro-chemical in character. The formation of insoluble zinc compounds and of soluble cadmium compounds appears to offer an adequate explanation of the greater intensity of attack on the cadmium-plated specimens.

As regards outdoor corrosion in an urban atmosphere, in the winter period the rain water in London consists of a very dilute acid solution. The formation of basic salts tends to the production of a partially protective coating on the zinc, whilst by contrast the products formed on cadmium are readily removed by rain, thus leaving the metal surface exposed to further attack. In the summer period, when the rain is relatively free from acidic constituents, it is interesting to note that the difference in the rate of attack of the two types of material is relatively small, only becoming acute as the winter period and its accompanying atmospheric pollution commences.

The results from the preceding research indicate that both cadmium and zinc form excellent protective coatings for appliances which are to be used for indoor work. The choice between the two coatings, always provided these are initially sound, can be made without any serious regard for their corrosion-resisting properties. Both should be durable and satisfactory. For outdoor exposure or for severe indoor exposure in highly saturated atmospheres there appears to be a strong case for zinc in preference to cadmium.

So far as the author has ascertained, cadmium is, however, largely used in practice for the first type of work, and as already stated, it is in that connection quite satisfactory. Finally, one would suggest that more should not be read into the foregoing research than is intended; much damage has been done both to the study of corrosion phenomena and to the faith of practical users of metal appliances by statements correct in their context, but gross exaggerations when mis-stated without the context.

#### The Institute of Metals Programme of Southampton Meeting

An interesting programme has been arranged for the autumn meeting of the Institute of Metals, to be held by invitation of the Mayor and Corporation at Southampton, from September 9–12. The proceedings will open on September 9 with the Ninth Autumn Lecture given by Professor D. Hanson, D.Sc. (Vice-President), on "The Use of Non-Ferrous Metals in the Aeronautical Industry." At 9.30 a.m. on the following day, a civic welcome will be given to the members by the Mayor of Southampton (Councillor Hector Young) in the Chantry Hall, after which they will spend three hours discussing metallurgical papers, several contributed by distinguished metallurgists from abroad. Visits will be paid to Portsmouth Dockyard (by steamer), Romsey Abbey and Winchester (by motor coach), the Supermarine Aviation Works at Hythe and Woolston, and the Ordnance Survey Office. In the evening there will be a reception and dance in the Royal Pier Pavilion by invitation of the Mayor and Corporation.

On September 11 the members will resume the discussion of papers at 9 a.m., the ladies meanwhile proceeding on a motor coach trip to Netley Abbey. At 12.30 p.m., the "business" side of the Conference will be formally concluded, and members and their ladies will be conveyed to R.M.S. "Mauretania," where they will be entertained to luncheon on board by invitation of the Cunard Steamship Company. Later there will be a series of visits, including an inspection of R.M.S. "Mauretania," and visits to the Floating Dock, International Cold Storage and Ice Co., Ltd., Harland and Wolff, Ltd., John I. Thornycroft and Co., Ltd., the Ordnance Survey Office, and Romsey Abbey and Winchester. In the evening a visit will be paid to the Empire Theatre.

On the concluding day of the meeting, a choice of two all-day excursions is offered—a motor trip in the New Forest, and a motor trip round the Isle of Wight. For the convenience of visitors who have the week-end free, and who desire to cross the Channel and return in an Atlantic liner, a trip to Cherbourg and back, with an extension to Dinard by motor coach, has been arranged. The party will leave Southampton at noon for Cherbourg in the "Mauretania," spending the night of September 13 in Cherbourg. On September 14, there will be a motor coach trip to Dinard via Coutances, Granville, Pontorson and Dinan, the night being spent at Dinard. On September 15, starting from St. Malo and passing through Paramé, the route will be by Mont St. Michel—where a stop will be made for lunch and to inspect the famous abbey—and thence via a number of interesting towns in Brittany and Normandy to Cherbourg, where the night will be spent. On September 16, the party will return to Southampton in the "Aquitania." The trip should be a particularly attractive one, as it can be made at an inclusive charge of £8 15s.

#### Dr. D. H. Ingall's New Appointment

DR. DOUGLAS HEBER INGALL, for the past two years head of the Constantine Technical College, Middlesbrough, has been appointed, subject to confirmation, principal of the Northern Polytechnic, Holloway, London. Dr. Ingall, who is 38 years of age, is a Doctor of Science, a Fellow of the Institute of Chemistry, and an Associate of the Institute of Physics. After acting as a metallurgist to a private company, he was appointed senior lecturer in metallurgy at the University of Birmingham, and in 1921 became the principal and the head of the metallurgical department of the Staffordshire County Technical College.

#### New Metallurgical Coke Works

An important development in the smelting of iron and steel is taking place at Pease and Partners, Roddymoor Colliery (Durham), where work has been commenced upon the construction of a new low ash plant to deal with coke produced at the colliery by-product works. The plant will include a conveyor, elevators, coal crusher and washing machinery.

Experiments have been carried out over a lengthy period, and as a result a new process has been evolved by which coke can be made with a percentage of ash less than four and a half. The new plant will be the only one of its kind in the North of England.

## Metallurgical Topics: Monthly Notes and Comments

### From Our Own Correspondents

#### Foundry Moulding Sands

RESEARCH Report No. 74 of the British Cast Iron Research Association on "The Properties of Some British Moulding Sands," by J. G. A. Skerl, is a publication which will do much to help practical men. It deals with the work of the few earlier writers who have treated the subject scientifically, but, as the author remarks, the literature of British moulding sands extends back only some twenty years or so, which shows to what an extent rule-of-thumb methods previously governed the industry. The methods of testing are described, the desirable properties of an efficient moulding sand being given as : (1) Strength; (2) permeability; and (3) Refractoriness. Modes of ascertaining each of these properties are detailed. They involve screening, sedimentation and elutriation for determining grain size and pore space, ramming, milling and strength tests, besides special permeability testing, for which the British Cast Iron Research Association has evolved a special apparatus. The conduct of refractory testing is also briefly described. The bulk of the paper is, however, devoted to describing the particular properties of various British sands, such as the Southampton sands, Erith sands, Pickering sands, Bromsgrove and Kingswinford sands, and the many other suitable deposits available to British foundrymen. The monograph is a valuable one on a subject the literature of which is all too scanty.

#### Molybdenum in Cast Iron

I. MUSATTI and G. Calbiani contribute to the June issue of *La Metallurgia Italiana* a research paper on special pig irons, devoted more particularly to molybdenum pig. Aluminium, besides serving to deoxidise cast iron, is said to favour the formation of graphite, and the authors indicate that an iron-aluminium alloy forms, containing 6 per cent. of aluminium. The views of Piwowarsky and Hurst on the function of titanium are quoted and confirmed, and the great improvement effected in the properties of cast iron by the presence of nickel are noted at some length. The main portion of the article is, however, devoted to an account of a research carried out at the Breda Institute, in Milan, on cast-iron containing molybdenum. The material used in the investigation was melted in a small electrical resistance furnace, and comprised a 4 per cent. carbon, low silicon pig iron; some 0.5 per cent. steel scrap containing as much as 1 per cent. of manganese, some ferro silicon ferrromanganese, and lastly, additions of 65 to 68 per cent. ferromolybdenum. The resulting casts had a total carbon content varying from 2.55 to 2.9 per cent. of carbon, with silicon from 1 to 2 per cent., low sulphur and phosphorus, about 0.5 per cent. of manganese, and a molybdenum content ranging from 0.28 to 3.19 per cent. The tensile strength varied from 50.5 kilogrammes per sq. mm. in an iron containing no molybdenum, but of otherwise average composition, to 59.6 kilogrammes per sq. mm. in the lowest molybdenum iron of the series, and fluctuated with little apparent relation to the molybdenum content, from 50.7 kilogrammes per sq. mm. to 84.5 kilogrammes per sq. mm. The Brinell hardness, on the other hand, rose fairly steadily with increasing percentages of molybdenum, from 199 to a maximum of 388, with the maximum molybdenum of the series (corresponding with 3.19 per cent. of molybdenum).

#### Influence on Heat and Corrosion Resistance

In another set of experiments tabulated, the carbon ran higher (from 3 per cent. to 3.36 per cent.), and the silicon was, on the average, lower. Much higher tensile strengths were obtained, but here again they do not appear to be related to the rather lower molybdenum contents, but to the carbon. The Brinell hardness figures rise with increasing molybdenum percentages, to 412. Broadly, the fractures of the specimens served to indicate that the presence of molybdenum greatly increases the fineness of the grain, and similarly the broad conclusion is arrived at that the mechanical properties of pig iron containing on an average 1.5 per cent. of molybdenum are greatly improved by such an addition. The behaviour of samples of these irons after exposure to a temperature of 800° C. for 24 hours showed that those

containing most molybdenum withstood the conditions best; a sample containing 1.54 per cent. of molybdenum, after this treatment, was quite devoid of scale or other surface indications of oxidation. On the other hand, and more especially the higher the percentage of silicon present, the lower was the corrosion resistance of the material in proportion as the molybdenum percentage increased. This low corrosion resistance is attributed by the authors to the finely divided graphite present, which increases the electrochemical activity of the material by forming minute electrical couples with the adjoining components.

#### French Researches on Corrosion

THE sub-committee of the Permanent Committee on Aeronautical Research of the French Air Department has issued the first portion of a Report on the Corrosion of Light and Ultra-Light Metals and Alloys, some of the findings of which may usefully supplement those of the very able reports on kindred subjects which have appeared in our own country under the auspices of the British Non-Ferrous Metals Corrosion Committee. The present investigations are by a number of different workers, and include a section on the investigation of corrosion tests generally by E. Herzog and G. Chandron; two by Cournot and Molnar, on the deterioration in mechanical properties, as the result of corrosion, and the investigation of the zone of activity of galvanic-couples, respectively; a research by Aubert and Prot, on the electromotive forces of solution, and one by Quillard on the investigation of methods of measuring the rise in temperature of activated aluminium alloys. In the first part of this report rolled aluminium and a wide range of aluminium alloys, as well as specimens of mild steel and of red copper (for purposes of comparison) were subjected to attack by dilute acid and the alkaline solutions and their tensile strength, elongation, and loss in weight subsequently determined. The experiments were in the first place directed to verifying the fact, previously recorded, that while corrosive action causes a decrease in the elongation it does not much affect the tensile strength of metals, apart from that accruing from the actual loss in weight. It was found that such effects as actually occurred could be grouped into two categories: (1) where the attack is uniform and the effects, apart from the actual loss of weight, are not marked in regard to mechanical properties, and (2) where the attack is local, in which case the elongation is very considerably reduced, yet, once again, the strength of the material remains practically constant. The specific behaviour in each set of circumstances, for the specimens of metals and alloys tested is embodied in tables which accompany this part of the report. The second portion, by Cournot and Molnar, gives details of experiments carried out by rather different methods, yet confirmatory of the earlier findings.

#### The Corrosion of Galvanic Couples

IN the third report, nine discs of aluminium and nine of duralumin had, at the centre of each, a rivet inserted, composed of one other metal in each case, the various metals for these rivets being respectively cadmium, zinc, iron, steel, aluminium, duralumin, brass, and red copper. They were immersed in artificial sea water and, generally speaking, aluminium was less attacked than duralumin in these circumstances, the former showing even corrosion in the vicinity of the couple. Zinc and cadmium both acted as protective metals, the latter being more effectual in this respect than zinc. Iron behaved very badly, particularly in contact with duralumin. In a further series of tests in actual sea water the discs were provided with a central copper rivet, and six additional ones placed concentrically around it. Both aluminium and duralumin were severely corroded, the former around the rivets, and the latter diffusively all over. A number of other exceedingly interesting tests are described in this section of the report, while in the succeeding section the results warrant the conclusion that very pure metals and very homogeneous alloys (compounds or solid solutions) resist corrosion better than heterogeneous metals; that heat-treated duralumin is more resistant than the merely annealed metal, and that very

homogeneous surface protection, devoid of porosity, is effective in preventing corrosion. Irregularities in heat treatment lead to irregular electromotive forces acting to the detriment of the metal and favouring corrosion.

### Structural Steelwork

NOTABLE examples of the recent employment of structural steelwork in modern buildings and bridges are contained in *Modern Steelwork*, a book published by the British Steelwork Association, Artillery House, Westminster, at a price of 5s., and edited by Mr. W. R. Gilbert. It forms at the same time a very interesting and informative summary of the latest achievements and a testimony to the reliability and adaptability of modern steel. A large number of photographs show buildings in various stages of erection and structural points of interest are illustrated by diagrams. Not the least telling is a series of six taken from the same point at different dates between December, 1927, and February, 1928, showing the two ends of the giant Tyne bridge approaching and meeting. One chapter, by Mr. Howard Robertson, deals with the erection of Imperial Chemical House, and there are others on skyscrapers, the reconstruction of bank buildings, fireproof flooring systems, welded steel pipelines, the modern Super-Cinema, steel rolling shutters, etc.

### Research on Minor Metals

ONE section of the report of Dr. W. Rosenhain on the work of the Department of Metallurgy and Metallurgical Chemistry at the National Physical Laboratory deals with research on the minor metals.

The attempt, he states, to produce beryllium of high purity by the decomposition of its iodide vapour in contact with a hot tungsten filament (method of van Arkel and de Boers) has been discontinued, at any rate for the present. The reason is that it has not been found possible to obtain a containing vessel which is not seriously attacked by the vapour of beryllium iodide. The vessels of hard glass, such as pyrex, etc., which had been used successfully in the production of other metals by this method, are attacked by the vapour of beryllium iodide, with the result that impurities such as silicon and boron are deposited upon the filaments together, with the beryllium metal, rendering the latter useless.

The line of attack which has been mainly followed is that of sublimation *in vacuo*. The sublimes produced by this method have not hitherto shown the ductility which it is anticipated that beryllium of high purity will possess. Careful microscopic examination has now revealed the presence in these sublimes of an appreciable amount of intercrystalline eutectic of a brittle character. Small particles which appear to be approximately free from this constituent have, however, shown indications that the metal, when ultimately produced in a state of purity, will prove to be ductile. Attention has accordingly been concentrated upon a thorough study of the impurities, particularly those of an intercrystalline character, which are present in the sublimate and in other forms of beryllium. This study has led in the first place to the conclusion that the sublimate, as hitherto produced, has become contaminated with beryllium oxide from the container upon which it was condensed. The view formerly held, that the presence of oxide in the sublimate was due to volatilisation of the oxide from the beryllia pot in which the molten metal has been contained, has been disproved, since it is found that when beryllia is heated alone *in vacuo* no appreciable loss of weight by volatilisation occurs. It seems probable, on the other hand, that the sublimate, when deposited in sufficient thickness upon the beryllia hood or container, comes under the action of the high-frequency induction field in the furnace and itself undergoes fusion, and then dissolves oxide from the container.

### Problems of Beryllium

THESE and other observations on the impurities present in beryllium indicate that the preparation of the metal in a sufficient state of purity to secure ductility offers a series of difficult problems. The solution of problems of this type, however, is not only of value with a view to the production of this particular metal in a state of high purity, but it is also likely to prove of importance in the metallurgy of many metals of a somewhat similar type.

Some further work has been done on the physical properties

and the metallography of cadmium. This metal appears to change appreciably on ageing at room temperature after previous cold working. The effect of this phenomenon on the general behaviour of the metal is being closely studied over a period of some years. After long ageing at room temperature, cold-rolled cadmium shows a considerable but gradual increase in grain size. The results of this research are being collected in the form of a paper, which should indicate some of the factors that are important in the study of soft metals at room temperature.

It has not been possible to make any progress towards the production of pure titanium metal, but some work has been done on the metallurgical uses of titanium in the form of the volatile tetrachloride. This compound offers the possibility of introducing titanium as an alloying element under circumstances which may prove to be advantageous for special purposes, and a beginning has been made in studying the effects of titanium chloride on various metals.

### Aluminium at Liege Exhibition

THE official opening of the International Aluminium Pavilion took place recently at the Liège Exhibition, and its inauguration is proving to be a popular and highly successful enterprise. The Bureau International de l'Aluminium, who are responsible for the arrangement and organisation of the pavilion, are receiving the co-operation of the prominent aluminium producers of Great Britain and the Continent and each country is well represented at the various stands.

The pavilion itself is constructed on the most modern lines, being both bold and simple in form. The centre of the ground floor has been devoted to a cinema where films illustrating the production and working of aluminium and its alloys are shown continuously. Around the hall are situated a number of stands exhibiting the more massive applications of the light metal industry. There is a demonstration of the various stages in the forging of a duralumin propeller from the billet to the finished article. Another stand exhibits a motor car chassis fitted with all-aluminium bodywork, and yet another an aluminium alloy lorry.

### Applications for Various Industries

On the second floor, the stands have been grouped under various industries; chemical, electrical, wireless, mechanical, automobile and textile industries each exhibit a representative selection of aluminium articles. A very fine collection of bright and matt, coloured, satin finished and embossed aluminium foil for decoration or packing purposes is shown; whilst household applications are represented by aluminium washing machines, refrigerators, kitchen utensils, etc.

The exterior of the building is picked out in polished or fluted aluminium strip, the windows are of duralumin, and the roof is composed of aluminium sheet. The aim of the organisers was, however, to construct not so much a house of aluminium, but to show in a few well-chosen examples, the ways in which the metal might normally be considered to be of use. Thus, the pleasing aspects of aluminium furniture, in the form of tables, chairs, desks and library, may be studied at leisure in the information bureau.

On the roof of the pavilion there stands an all-metal aeroplane of duralumin, and to complete the scheme of organisation, the basement reveals a room where welding and machinery is demonstrated to those interested.

### Iron and Steel Statistics

THERE is little satisfaction to be gained from the latest issue of the Statistical Bulletin of the National Federation of Iron and Steel Manufacturers, which shows that during June the number of blast furnaces in operation in the United Kingdom declined by 8 during the month to 133, and the number of furnaces blowing was lower than at any time since the end of 1928. Production of pig iron amounted to 563,200 tons compared with 614,500 tons in May and 657,800 tons in June last year; production in June last year, however, was still on a rising curve, whereas production this year has consistently declined. Steel production in June amounted to 600,100 tons compared with 691,900 tons in May and 830,900 tons in June last year. The total exports of iron and steel in the first half of 1930 amounted to 1,797,500 tons compared with 2,240,800 tons in 1929.

## Trade, Commerce, Finance : The Month in Review

From Our Market Correspondent

THE experience of the past month in the iron and steel trade, and the prospects for the weeks ahead, are alike discouraging. There has been no relief to the dull depression which has prevailed during recent months, and which becomes more serious as time goes on. The markets are lifeless and almost without hope. One feels that everything practical, inside the industry, has been tried that might restore some measure of prosperity, and that there is nothing left but to continue the struggle as gamely as possible until something—one does not know what—arrives to turn the scale. A short time ago there was a lively expectation that after all safeguarding would soon be applied to the steel trade. No doubt the bankers' manifesto was largely the cause of this; and its immediate effects were seen most clearly in the industrial section of the stock exchanges. There is now such an overwhelming weight of support for the abandonment of the policy of free imports that it is difficult to understand why no official move is made. But changes of this kind are very slow in coming, even when their need is recognised; and it is to be feared that conditions will become considerably worse than they are now before the Government can be induced to do what ought to be done. The refusal to publish the report of the inquiry into the condition of the iron and steel trade is no doubt due to the fact that the report would definitely advocate some form of protection, and it requires a great deal of courage—more than has yet appeared—to make such a drastic reversal of policy. Perhaps something may happen when the unemployment figures are well over the 2,000,000 mark, as they are soon likely to be.

### Continental Underquoting

Added force will be given to the demand for safeguarding by the recent news from the Continent. The International Steel Cartel has at last broken down, as far as the fixing of selling prices of finished steel is concerned. The depression in the steel industry in Germany and Belgium has been too severe a test for the strength of the organisation, which has proved unable to endure in face of the deliberate underquoting which has been resorted to, particularly by Belgium. How often has such an experience been repeated! Associations or agreements which are formed merely for controlling selling prices carry on very well during periods of good or fair trade, but they almost invariably fail under the strain of prolonged bad trade. To make them effective through good and bad times the control must be more thorough and wider in its scope than the mere fixing of prices. It is too early yet to gauge the full effect of this turn of events abroad, but one thing is certain: until a fresh agreement is reached there will be an outbreak of cut-throat competition for the English market so long as that market remains unprotected. Already small steel bars are being offered from the Continent at prices below those of the billets from which they have to be rolled! Naturally such a position cannot be maintained indefinitely. Either billets will fall in proportion, or the price of bars will be advanced, and just now these bars are being offered at prices ranging from £5 10s. to £5 15s. per ton, delivered into the Birmingham district, that is at least £2 per ton below the home prices. There is a similar margin between the prices of other finished products, and if this wide divergence persists for any length of time it will be a severe temptation to some of the consumers in this country who have hitherto refrained from using Continental steel. How can such an abnormal condition be met except by some form of protection?

From still another quarter pressure is likely to be brought to bear on the Government. The recent publication of the comparative wages costs in the steel industry in Great Britain, Germany, France and Belgium has shown how unfairly handicapped the British steel makers are, when in competition with the Continental works. Unless some means of meeting this situation is forthcoming there must of necessity be a reduction in the standard of living of the men employed in our steel works either through a fall in their nominal wages or a reduction in the real wages owing to short working. The men's leaders will resolutely oppose any official reduction in wages, and they have therefore no alternative but to give vigorous support to the only other method of overcoming this menace from abroad.

### Rationalisation in Scotland

The latest addition to the rationalisation schemes amongst the steel works is that reported from Scotland, the taking over of James Dunlop and Company by Colville's. Dunlops have a well established reputation as plate makers, and their plate mills are to be closed down and the trade transferred to Colville's, who will thus secure a much larger output for their own mills. It will not be surprising if this process is extended in Scotland until eventually there is only one steel producing group in that country. By eliminating surplus plant and the concentration of production in the modern mills the steel trade there will be in a much stronger position. We have had plenty of the rationalisation amalgamations of which the constituent firms have been too weak to stand alone and in which the members are mainly occupied in holding each other up. Their last state may prove to be worse than the first.

### No Reserve of Orders

Just now the holiday season is in full swing, and will continue through the month of August. Steel makers are not worrying about the holiday stoppages, extended as they are likely to be. These intervals will be welcomed as an opportunity of building up something of an order book. Orders are coming in so slowly that there is no certainty of continuous operation; indeed most of the mills are working only part of the week without any reserves of orders to carry forward. Delivery of any kind of finished steel, plates, sections or bars can be obtained immediately the order is placed. Dorman, Long and Co. have closed their Port Clarence steel works because of the depression in trade and will not re-start them until there is a decided improvement. This means unemployment for about 2,000 men. For a similar reason one of the Guest Keen plants in South Wales is being shut down. The Ebbw Vale steel works are doing practically nothing. In Lincolnshire also the blast furnace plant of the North Lincolnshire Iron Co. has been closed down. On the North East Coast furnaces have been blown out owing to the lack of orders. And so the tale goes on. It is a most dispiriting situation, with no improvement in view. What the autumn will bring it is not wise to prophesy, but there is no ground for optimism. An indication of the state of trade was given by the recent Birmingham quarterly meeting, which was one of the smallest on record, and the amount of business done was equally small.

### Pig Iron Prices

During the past month pig iron prices have changed considerably. The West Coast hematite makers last week reduced their prices for special irons 2s. per ton in the hope of encouraging buyers to come into the market. There has been a gradual curtailment of output owing to the falling off in demand, and still the shortage of orders left an excess of production. Whether this latest fall in price will have any good result is doubtful as buyers seem to maintain their wait-and-see attitude. The Cleveland pig iron makers are finding it extremely difficult to maintain either their prices or their outputs. In the Midlands the Central Pig Iron Producers' Association, which includes all the makers, made a reduction of 2s. 6d. per ton in all classes of iron about a fortnight ago. Buyers, however, were looking for a concession of about 5s., and they are still not offering much business. The prices of finished steel controlled by the Associations remain unchanged, and will remain so at least until October, as there is no joint meeting to be held before then. In the outside materials, such as boiler plates and small bars, competition continues to be very severe. The small bar rollers particularly are likely to suffer still further from the Continental competition. Unfortunately, the motor trade is passing through an extremely dull period, more so than usual at this time of the year, and this too is adversely affecting the trade of the bar mills.

The production figures of the month of June show a further decline, which is not altogether due to the Whitsuntide holidays, and there was a reduction of eight in the number of blast furnaces working.

**Some Inventions of the Month****By Our Patents Correspondent***Abstracts of other Patents of metallurgical interest will be found in our Patent Literature published weekly in THE CHEMICAL AGE.***Alloys**

TIN-LEAD alloy plating which may be easily soldered is obtained by electrolysis of a bath containing the tin and lead salts of benzene-1:3-disulphonic acid. A tin-lead anode is used, and by adding cadmium salts to the bath a deposit of particularly low melting-point may be obtained. A bath is specified containing per litre of water 100 grams each of the tin and lead salts of benzene-1:3-disulphonic acid, 20 grams of cadmium perchlorate, 50 grams of benzene-1:4-disulphonic acid, 5 grams of gelatine, and 5 grams of phenol. See Specification 329,346, dated February 16, 1929, by M. Schlotter, of Berlin.

A NON-CORRODIBLE alloy described in Specification 329,361, dated January 16, 1929, by W. Haddan, of London, and J. Winfield, of Leicester, contains copper and aluminium and 0.25-0.4 per cent. of manganese, together with nickel or iron or both. Permissible proportions are 88.5-90 per cent. of copper, 7.5-10 per cent. of aluminium, 1 per cent. of nickel and up to 2.75 per cent. of iron. Up to 1 per cent. of tin, lead, or similar metal may be added in some cases.

**Alloy Steels**

TITANIUM steels containing not more than about 1 per cent. of carbon and 0.8 per cent. of titanium, the ratio of carbon to titanium not exceeding about 5:4, are described in Specification 329,705, dated February 25, 1929, by W. and H. Mathesius of Charlottenburg, Berlin. The proportions of carbon, silicon, and titanium must be such that the carbon is substantially wholly combined with the titanium in the form of carbides in solid solution. Manganese or chromium may be present to the extent of about 0.5 per cent. each, and small amounts of boron, zirconium, tungsten, molybdenum, vanadium, or uranium may be added. The steels may be made by the method of Specification 221,529, or by adding carbon-free ferro-titanium to molten iron containing about 0.1 per cent. of carbon deoxidised with carbon-free silicides or to carbon-free molten iron deoxidised with silicides containing some carbon.

**Cadmium Plating**

A DRY preparation for use in compounding an acid cadmium plating bath comprises a mixture of an acid sulphate, a cadmium compound capable of reacting therewith in aqueous solution to produce cadmium sulphate, ammonium sulphate, and a brightening agent. Cadmium oxide, hydroxide, carbonate, sulphate, or chloride may be used. Glue, gulaç, shellac, natural resin, isinglass, and agar-agar are specified as brightening agents. See Specification 328,574, dated January 29, 1929, by C. H. Humphries, of Indianapolis, Indiana, U.S.A.

**Copper and Zinc**

In a process for treating lyes containing copper and zinc e.g., lyes obtained by leaching chloridised roasted pyrites, the copper is partially reduced to cuprous chloride by treatment with sulphurous acid, with addition of chlorides if necessary, until a solution containing a suitable concentration of acid, e.g., 5-10 grammes of hydrochloric acid per litre, is obtained. The reduction is then completed by means of finely divided copper. Gold and silver present may also be precipitated by addition of excess of copper. The solution and the precipitate after the treatment with copper may be treated with iron to convert the cuprous chloride into metallic copper. See Specification 329,225, dated July 13, 1929, by N. E. Lenander and I. Ryen, of Lokken Verk, Norway.

**Separating Iron from Cobalt, etc.**

SOLUTIONS containing cobalt and rich in iron, and in some cases containing also zinc and copper, are obtained by lixiviating chlorinated roasted pyrites, and are then treated for separating the iron. Scrap iron is first added to precipitate copper, and sodium sulphate may be removed by cooling to 10° C. An alkali metal chlorate, preferably sodium chlorate, in amount equal to about  $\frac{1}{2}$  part by weight of the iron present, is then added, the temperature being kept at about 60° C. In about an hour the iron is oxidised, and is then precipitated by lime. Sulphur present is also precipitated, partly as basic iron sulphate and partly as gypsum. Cobalt, zinc, and other valuable constituents are recovered from the filtrate. See

Specification 329,543 (Potts), dated June 18, 1929, a communication from Orkla Grube-Aktiebolag, Trondhjem, Norway.

**Platinum**

METALS of the platinum group are recovered from ores, concentrates, etc., containing chromite by oxidizing the material to form chromates and ferric oxide and smelting the product either (1) by a lead blast furnace method or (2) with matte-forming materials as described in Specification 316,063 (See THE CHEMICAL AGE, Vol. XXI, p. 23 [Metallurgical Section]). If method (1) is adopted, the material may be roasted in a reverberatory furnace with lead oxide, preferably with addition of lime, the roasted product being then briquetted or sintered with silica and smelted in a blast furnace to obtain lead containing the platinum group metals and an iron-lime silicate containing the chromic oxide. If method (2) is adopted, the material may be roasted with alkalies, the sodium and calcium chromates being then leached out of the roasted material, and the residue being smelted with silica and matte-forming materials to collect the platinum group metals in a matte. See Specification 328,564, dated January 28, 1929, by A. R. Powell, of Amersham, Bucks, E. C. Deering, of New Barnet, Herts, and Johnson, Matthey and Co., Ltd., of London.

**Zinc**

A PROCESS for agglomerating zinc ores prior to smelting is described in Specification 328,162, bearing the International Convention date June 7, 1929, by Soc. Anon la Nouvelle Montagne, of Engis, Belgium. A powdered mixture of raw blende and pre-roasted blende is sprayed with water or with a solution of zinc sulphate or dilute sulphuric acid to form granules which harden owing to the formation of zinc oxy-sulphate therein. The preferred method is to expose the mixture on a moving surface in a succession of superposed layers each of which is sprayed separately before receiving the next layer. The granules may be dried in admixture with some of the powdered material.

**Commercial Intelligence**

*The following are taken from printed reports, but we cannot be responsible for any errors that may occur.*

**Mortgages and Charges**

[NOTE.—*The Companies Consolidation Act of 1908 provides that every Mortgage or Charge, as described therein, shall be registered within 21 days after its creation, otherwise it shall be void against the liquidator and any creditor. The Act also provides that every Company shall, in making its Annual Summary, specify the total amount of debt due from the Company in respect of all Mortgages or Charges. The following Mortgages and Charges have been so registered. In each case the total debt, as specified in the last available Annual Summary, is also given—marked with an \*—followed by the date of the Summary, but such total may have been reduced.]*

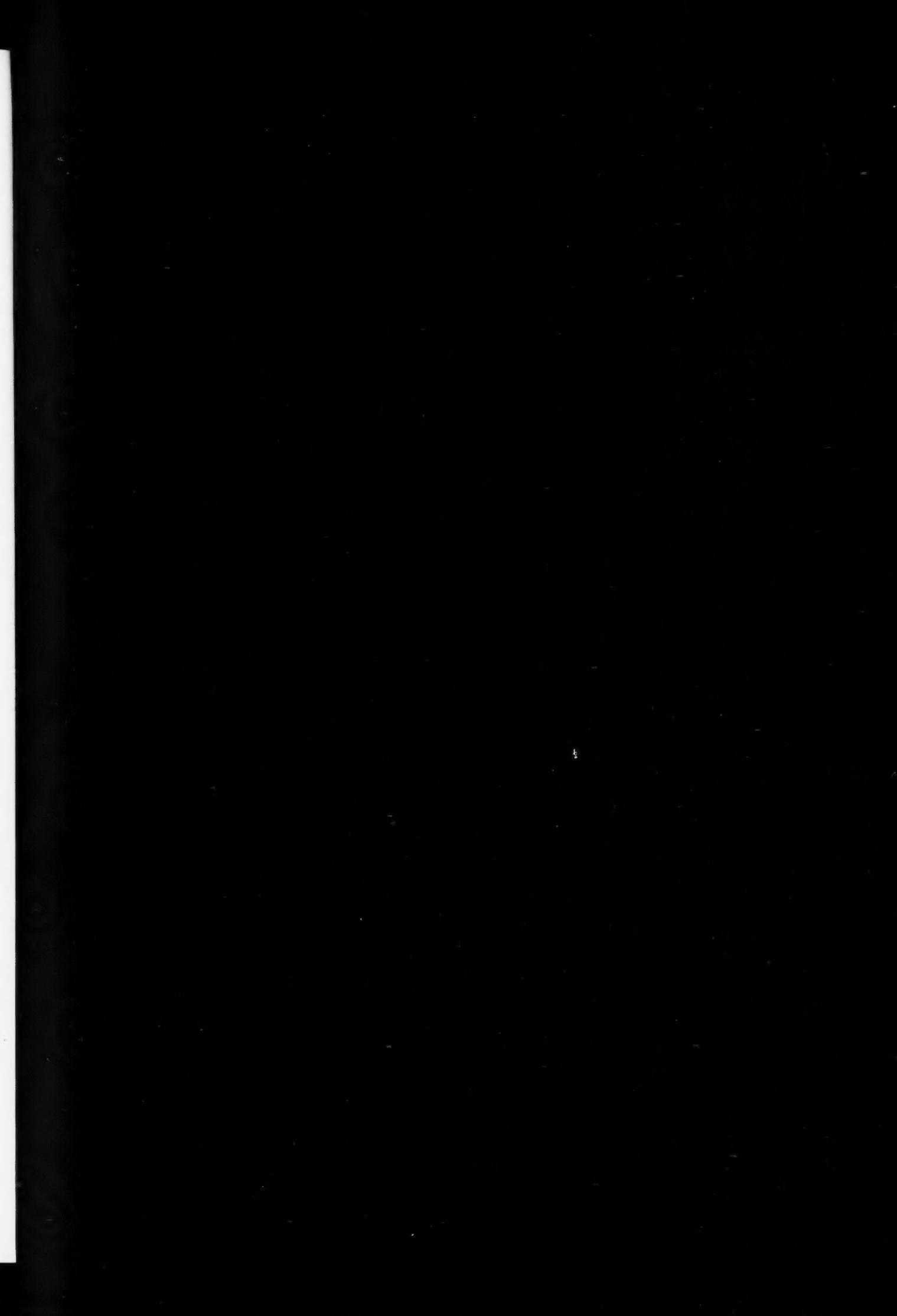
ALUMINIUM CORPORATION, LTD., London, S.W. Registered May 28, £250,000 first debenture stock and premium of 5 per cent. (secured by trust deed dated May 16th, 1930), present issue £200,000; charged on properties at Dolgarrog, etc., also general charge. \*£1,123,465. December 2, 1929.

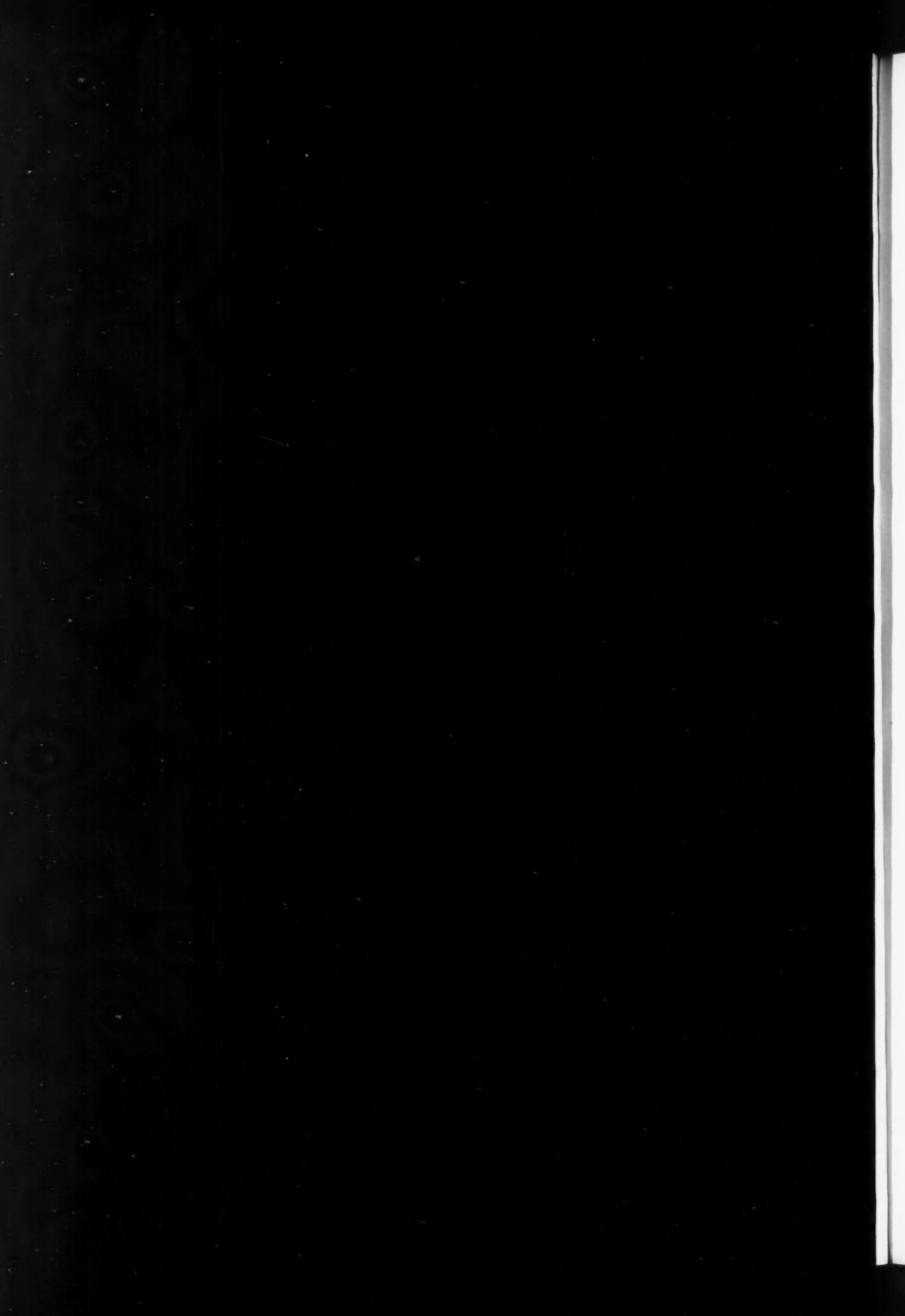
SKELLINGTONS, LTD., Sheffield, iron and steel manufacturers.—Registered July 12, £12,000 first debenture to Branch Nominees, Ltd., 15, Bishopsgate, E.C.; general charge; also reg. July 12, series of £5,000 debentures present issue £1,500; general charge. \*£10,000. April 11, 1930.

WHITWORTH AND JONES, LTD., Coventry, manufacturers of alloys. (M., 12/7/30.) Registered June 24, mortgage to National Provincial Bank, Ltd., securing all moneys due or to become due to the Bank; charged on properties in St. Mary's Road, Leamington, etc. \*Nil. December 30, 1929.

**Satisfactions**

BEARDMORE (WILLIAM) AND CO., LTD., London, S.W., steel manufacturers—Satisfactions registered July 14, £1,000,000, registered August 30 and September 26, 1904, and August 6, 1927; £26,532 16s. 6d. part of amount registered August 9, 1915; £720,000 registered December 1, 1921, £600,000 (not ex.), registered July 11, 1924, March 26, 1925, and September 24, 1928; £250,000 registered November 26, 1924, £50,000 (not ex.) and £50,000 (not ex.) registered September 2, 1925, and £600,000 (further security) registered September 24, 1928.





## Monthly Metallurgical Section

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**NOTICE.**—Communications relating to editorial matter for our Monthly Metallurgical Section should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.

### International Trade in Manganese Ore Increased Production and Its Effects

Some interesting features of the international trade in manganese ore during the first quarter of 1930 are considered in the following article by Daniel P. Lloyd, of the Minerals Division of the U.S. Department of Commerce, published in a recent issue of "Commerce Reports."

SEVERAL factors make international trade in manganese ore of particular significance. From 92 to 95 per cent. of the total consumption of manganese is utilised in the production of steel, yet not one of the more important steel-producing countries of the world produced more than a small part of its total requirement of manganese. Although manganese is known to occur in practically all parts of the world, the principal producing countries for many years have been Russia, India and Brazil, with the Gold Coast of South Africa added since the war. Of these countries only Russia produces steel to any extent, and its production of manganese is far in excess of its requirements in the steel industry. Thus, international shipments of manganese ore in the past have been largely from these four producing countries to the United States, Germany, France, Great Britain and Belgium-Luxemburg.

#### Effect of Larger Stocks

During the first quarter of the present year shipments of ores were unusually large, in fulfilment no doubt of earlier orders, while the manganese market has been in a decidedly depressed condition. Without exception the principal consuming countries imported more ore than in the corresponding period last year, and much more than their average normal requirements. As far as determinable, however, a large part of this import has gone to replenish stocks rather than into actual consumption, so that it would seem that receipts have been above actual consumptive requirements. The manganese market undoubtedly for some time will feel the effect of this increase in stocks as well as the decrease in steel production throughout the world, and little evidence is now available to indicate any rapid change in the market situation.

Large imports of manganese into France have formed a feature of the international trade for several years, and the first quarter of the present year was no exception. As the French steel output is relatively small, and its production of ferromanganese by no means commensurate with such large imports, there is considerable interest as to the use made of so much ore. Details of the actual consumption of manganese per ton of steel made in France are not available, but the ratio between the manganese content of net imports of manganese ore and ferromanganese to steel produced is considerably higher than in other countries. In 1929, for instance, net imports were equivalent to 85.3 lb. of metallic manganese per ton of steel produced.

The following table shows imports into the principal consuming countries during the first quarter of 1930, with comparative statistics for the corresponding quarter of 1929:

| Imported into                             | First quarter of— |              |
|---|-------------------|--------------|
|   | 1929              | 1930         |
|   | Metric tons.      | Metric tons. |
| France .....                              | 155,228           | 207,372      |
| United States .....                       | 104,601           | 169,480      |
| Germany .....                             | 58,203            | 96,768       |
| Great Britain .....                       | 53,845            | 89,021       |
| Belgium-Luxemburg (first two months only) | 41,273            | 44,275       |

#### A Moroccan Development

Another important factor in the French position, and one which may have a pronounced effect upon the future world manganese market, is the present development of exploita-

tion of manganese deposits in Morocco. The ore is reported to average about 45 per cent. manganese, with pockets of dioxide of manganese. A railroad is under construction which will connect the Bou-Arfa mines with the port of Nemours, through Oudja. Completion is expected during 1931, when exports are to begin from stocks already mined.

#### Russian Production

Russia apparently has succeeded in capturing the greater part of the United States market for manganese ore. Imports from that source increased more than 60,000 tons over receipts of the 1929 quarter, and accounted for 54 per cent. of the total. India is the only country to show any marked decrease in shipments. Not only are Indian producers finding it difficult to compete with the Russian and Gold Coast manganese in the United States market, but agreements made between Indian mine owners and British producers of ferromanganese are tending to make India a closed market. That is perhaps the principal reason why exports from India to the United Kingdom almost doubled in 1929. Another interesting observation is the sharp increase in imports from the Gold Coast. It is reported from Accra that, given good market conditions, shipments from this source in 1930 may be expected to exceed those of 1929, when shipments reached the record total of 408,224 tons of high-grade concentrate.

Germany's imports increased considerably during the quarter, receipts being approximately 65 per cent. above those for the corresponding quarter last year. Russia and British India furnished approximately two-thirds of the shipments. It is probable that those credited to Australia are largely reshipments from Java or British India.

#### Present Position of the Market

From the standpoint of the producer there is little encouragement in the present position of the world manganese market. Steel production is down; consumers seem to have covered their requirements for the time being, and stocks are mounting in the principal consuming countries of the world. Competition between producers has forced prices down to the lowest post-war level, and few orders are being placed at any price.

Among the factors which have led to this situation none has been discussed so frequently as the influence of Russian production and Soviet policy. According to official Union of Socialist Soviet Republics statistics, exports from Russia during the fiscal year ended September 30, 1929, were 900,000 tons, compared with 499,000 tons in the preceding year, and every effort is being made to increase exports similarly this year. The natural effect upon a market of such a large increase in supplies is obvious, but the result has been doubly depressing owing to the determination of the Soviet to control the world market by effecting sales at any price. The result has been disastrous to many of the producing mines in other countries, some of which have been forced to close down, while others are able to survive only by practising the greatest economies. In India the situation is being met by the introduction of more modern machinery in order to increase the efficiency of the mines. The Government is co-operating by spending large sums of money to develop the port of Vizagapatam, approximately midway between Madras and Calcutta, which will reduce the rail haul for an important group of mines. The improvements being made in the Gold Coast have already been mentioned.

No account of the industry would be complete without reference to the intensive development taking place in the Posmasburg area of South Africa, where, it is claimed, are very rich and extensive deposits of manganese. The chief obstacle to their exploitation has been the absence of railway communication with the coast. During 1929 the Government undertook the extension of the railway to these fields and it is expected that this line will be completed in June of this year. In the meantime the Manganese Corporation (1929), Ltd.,

which controls the mines, has been busy in opening up the deposits and erecting plant equipment. At present over 1,000 natives are employed in construction and mining, and the daily production of high-grade ore is said to approach one thousand tons.

With the entrance of manganese ore from this and other new sources into the world market, it is not anticipated that steel producers will have any difficulty in obtaining their requirements of manganese for some time to come.

## Nickel Plating for Mass Production

By N. R. LABAN

*A paper of considerable interest to nickel platers who require to work at high current density was recently read before the Electro-platers' and Depositors' Technical Society by Mr. N. R. Laban. The difficulties involved in deposition at high current density are reminiscent of those encountered in chromium plating, but, in Mr. Laban's view, the advantage obtained is not merely one of speed but of better quality deposits as compared with nickel plating at low current density.*

Mr. LABAN, in the course of his remarks, said he intended to describe the methods which have proved highly satisfactory in production over a period of several years for the deposition of nickel. The methods are particularly suitable when the nickel is used as a basis for chromium. The following rough classification is adopted:—Low current density—up to 25 amp./ft.<sup>2</sup>; medium current density—25 to 50 amp./ft.<sup>2</sup>; high current density—above 50 amp./ft.<sup>2</sup>. Current densities between 60 and 100 amp./ft.<sup>2</sup> are quite a practical proposition for the majority of the components used in motor and cycle work. The advantages in the use of high current density are not confined to output, but the deposits obtained possess excellent mechanical properties, admirably suited to engineering purposes. Any plant which is capable of giving good results at, say, 30 amp./ft.<sup>2</sup> can be made to operate at high C.D. with slight modifications.

The substitution of electrolytic cleaning for hand-scouring processes is necessary before the full advantages of medium or high C.D. deposition can be appreciated, and, except in special cases, it should be unnecessary to employ labour in scouring. The first operation is the well-known alkaline cleaning, in which the work is made cathode on a 5 or 6 volt supply. Depending on the type of polishing medium employed, and on the length of time the work is allowed to stand after polishing, cleaning is effective in from 5 to 30 minutes. In cases where the cleaning is slow, arrangements may with advantage be made to reverse the polarity during the cleaning process. It will also be found advantageous to agitate the cleaners and swills by means of compressed air, as this prevents accumulation of scum on the surface.

### Anodic Acid Cleaner

The electrolyte consists of strong sulphuric acid containing about 3 oz./gallon of potassium bichromate. The lead lining of the vats is made cathode on a 6 volt supply, or, if preferred, lead sheets may be used as cathodes insulated from the vat lining. The cleaner has proved very satisfactory for the great majority of steel components, and is a complete guarantee against stripping of the nickel deposit during chromium plating. It can be shown by simple tests that the nickel-steel junction is stronger than mild steel.

The placing of the work on the anode bar completes the circuit, and for a few seconds a heavy current passes (up to 100 amp./ft.<sup>2</sup>), rapidly falling as the steel becomes passive until, after about 30 seconds, a steady current of about 20 amp./ft.<sup>2</sup> passes. The oxygen evolved from the work performs the triple duty of oxidising the grease to non-greasy organic matter, rendering the steel passive and exerting a scouring action. After half to one minute in the cleaner, the articles are removed and transferred to the swills.

### Few Cases of Stripping

The steel surface is found to have a dull satiny surface, the brilliant polish being completely destroyed. A perfect "key" is provided for the deposit, and the fine etch obtained does not interfere with the final nickel mopping. The acid is maintained between 50° and 55° Bé. More or less than this is liable to produce severe etching, which is apparent in the finished work. As an illustration of the effectiveness of this cleaner, the percentage of work stripping during chromium plating averages 0.5 per cent., the output being 12,000 com-

ponents per week. If cases of stripping do occur, they are mainly due to double deposits. In production, an anodic cleaner of about 100 gallon capacity will handle the same quantity of work as may be expected from three scourers, and has the advantage that unskilled labour may be employed.

### Importance of Swills

The importance of swills after acid cleaning needs no emphasis. On the efficiency of the swilling and draining operations depends to a great extent the variations in P.H. value of the plating solutions. Two separate swills, one after the other, have been found to answer fairly well, both being supplied with compressed air for agitation and with a good supply of running water. All articles which are likely to retain quantities of acid must be turned over and drained after each swill. The passivity of the steel persists for five minutes or more, and there is no harm in allowing the articles to drain or to stand in the second swill water for a minute before transferring to the plating vat. The first swill may be expected to contain up to 10 per cent. of acid during normal working, the second swill, therefore, functions as an ordinary swill after a 10 per cent. acid dip.

Plating vats are of the usual lead-lined wood type, but at all costs the use of an inner wood lining is to be avoided, as the high temperatures necessary extract sufficient organic matter even from well seasoned wood to limit seriously the current density that can be employed. Vigorous agitation is essential unless a moving cathode is employed, and it is important that the air stream should envelope all the components. Continuous filtration is also an essential. For a 400 gallon vat, if the anodes are bagged, one filter should be sufficient. If the anodes are not bagged, two filters are inadequate.

To exceed 70 amp./ft.<sup>2</sup> with success, some form of moving cathode is essential. If the work moves through the solution at a speed not less than 12 feet per minute current densities of 100 amp./ft.<sup>2</sup> are possible on many components. In this case the agitation need not be so violent; it is, in fact, surprising how little agitation is required, and it is sufficient if each article passes over the air stream at intervals during its travel.

### Voltage

The voltages required for nickel plating with the solution given later and 11 inches electrode distance, are as follows:—Low current density—up to 3 volts; medium, up to 3 to 5 volts; high, up to 5 to 10 volts. The best arrangement is for each vat to be operated by a separate 10 volt motor-generator, and it must be borne in mind that for a 400 gallon vat with double cathode bar working at 70 to 80 amp./ft.<sup>2</sup> on average sized components a current capacity of 1,000 amperes is not excessive.

The standard single salt solution, containing not more than 2½ lb./gallon of single nickel salts, and 3 oz./gallon boric acid is used. For the conducting salt, either nickel or sodium chloride is satisfactory. Actually, high current density work is quite practicable with a  $\text{NiSO}_4$  content of 1.8 lb./g. and medium current density work with as low as 1/5 lb./g., but these figures are too close to the limit for production. In practice the solution should be made up to 2.5 lb./g. as soon as the concentration reaches 1.8 lb./g. The density is between 15° and 17° Bé.

There is much discussion as to the extent of technical control necessary to maintain the  $P_h$  value between safe limits. It can be safely said that acidity variations which are produced in the nickel vat itself are comparatively slight, even when working at a current density of 80 amp./ft.<sup>2</sup>, and that most of the trouble in this direction comes from outside. The efficiency of the single salt-boric acid solution is very nearly 100 per cent., and little variation in  $P_h$  is to be expected. Between 5·0 and 6·0 is the useful range from the point of view of the practical plater, although quite sound deposits can be obtained between  $P_h$  4·0 and 6·5. The adjustments of acidity are, of course, made with nickel carbonate.

The solution temperatures required are as follows:—Low current density, 25 to 35° C.; medium, 35 to 50° C.; high, about 50° C. The best results when working at a C.D. above 70 amp./ft.<sup>2</sup> are obtained at 60° C., and the highest possible C.D. combined with a high temperature gives the most satisfactory deposits.

#### **Loss in Throwing Power**

One of the greatest difficulties of high C.D. deposition is the loss in throwing power. This may be so serious as altogether to preclude the use of a C.D. above 40 amp./ft.<sup>2</sup> on certain components. To secure an adequate deposit on recessed portions of the article, it may be necessary to give a far longer time in the vat than is required to deposit 0·001 in. The more prominent surfaces therefore receive a great excess of nickel. If this is continued on production day in and day out, the wastage of nickel becomes serious, and it would be far better to reduce the C.D. This matter must be left to the discretion of the plater, since obviously the practice should be, not to

place at a given C.D. for a given time, but to make certain that recessed portions receive a satisfactory deposit. If this can be done by depositing at 100 amp./ft.<sup>2</sup> for 10 minutes, there are no objections to so doing.

To plate a cycle handlebar at a C.D. of 80 amp./ft.<sup>2</sup> requires 55 amperes. Similarly, an average radiator shell would require 320 amperes. It is obvious that simple methods of wiring are quite unsuitable, and substantial jigs or hooks made from solid brass or copper are necessary. Furthermore, it is essential to secure firm contacts as a break in the current of a fraction of a second is sufficient to produce a double deposit and consequent stripping during chromium plating.

Fairly heavy articles of not too large a surface area may be suspended on hooks, their weight preventing broken contacts. On the other hand, if the article has a big surface, the current passing may be sufficient to heat up the point contacts under the solution and result in double deposits. It is useless to place light articles on hooks or suspenders; they should be wired, on frames, so that the whole frame can be carried through the various swirls and cleaners. Great care must be taken not to wire up too tightly otherwise a bare patch will be found under the wire.

In general, as much consideration must be given to the preparation of a load of work to be plated with nickel at high C.D. as would be taken with chromium plating, the special points to be watched being:—Screening, loose contacts, adequate thickness of wire, or rods, efficient agitation. The use of jigs of heavy wire or strip involves losses of nickel due to deposition on the jigs. There is need of a lacquer or enamel which will withstand repeated immersions in hot alkali and strong sulphuric acid.

## **Metallurgical Topics: Monthly Notes and Comments**

### **From Our Own Correspondents**

#### **The Projected Copper Refinery**

ALTHOUGH the reports which, during the last few weeks, have been current respecting the project for putting down a large copper refinery in this country, to refine Northern Rhodesian copper, conflict somewhat as to details, there is little doubt that such a project, backed by important Northern Rhodesian interests, is under serious consideration. It will probably be built on the Mersey, and a site near Widnes is said to have been selected for the new works. At the same time a good many misconceptions prevail as to what precisely is under contemplation. To begin with, the project does not involve, as some of the papers have erroneously reported, any scheme for smelting copper ores; the freight charges would preclude anything of the sort being profitably undertaken. The works will refine copper, smelted at Rhodesian works. The chief interests behind the project are those of the Bwana M'Kubwa Copper Mining Company, whose chairman, Sir Edmund Davis, referred to it at the annual meeting held about a fortnight ago. Even at present prices, copper mining can be undertaken more profitably in Northern Rhodesia than at many copper mines in North America, and smelting operations conducted more profitably, seeing that labour, although not plentiful, is still cheap. As is well known, the mineral resources (copper, zinc, vanadium, and even tin) are enormous, although so far as tin is concerned the Nigerian resources are far more extensive.

#### **An Example from Belgium**

DEVELOPMENTS in Northern Rhodesia have proceeded, however, at a far slower rate than in the adjoining Belgian Congo territories. The project of building a refinery in England to deal with Rhodesian copper resembles one to which effect was long ago given by the Union Minière du Haut Katanga, which has a very large works at Hoboken, near Antwerp, where copper from their great works at Panda and Lubumbashi are refined, and high-grade electrolytic copper (99·5 per cent. Cu) is produced, as well as "best selected" running to 99·8 per cent. of copper. For the production of electrolytic copper alone the plant comprises 832 electrolysing vats, each housing 30 copper anodes, and the copper output capacity of the works is 80,000 tons per annum. At Oolen, also belonging to the Union, but administered, like

Hoboken, by its subsidiary, the Société Générale Métallurgique de Hoboken, tin from Kikole and Kayumbo ores is smelted, although the most important, and probably the most profitable, of the Hoboken activities is the extraction of radium from the pitch-blended ores of Chinkolubwe, Katanga, of which during 1929 the production attained to 60 grammes. Finally, the Oolen works are by far the largest producers of cobalt in the world, the 1929 output having been equivalent to 708 tons of that metal, either as metal or as cobaltous salts, whereas Canada produced no more than 300 tons.

While it is too much to expect that the British copper refinery projected for Lancashire should emulate all the activities of the Belgian enterprise, which possesses unique resources, Bwana M'Kubwa, Broken Hill, and other Rhodesian interests ought to be able between them to establish, in the home country, a works that could produce profitably much of the copper, zinc, vanadium and other products in respect of which we are far too dependent upon other nations at present. If in Belgium and in Germany, where works carrying out similar operations have proved so successful, the metallurgy of essential base metals can be profitably conducted and employ thousands of workpeople, similar works in England, drawing on the vast Empire resources at our disposal, should at least serve in some measure to help to decrease our growing unemployment, unless, as may unhappily result, our labour costs still continue to prove a hopeless handicap.

#### **Accelerated Fatigue Tests**

In these days the ascertainment of the endurance limit under fatigue stresses of metals used in engineering construction is of extreme importance from two severely practical and industrial points of view, apart from all the more purely scientific and academic considerations of how and why metals fail. Absolute results can, in all probability, be established only by long-time tests; accelerated tests are always open to criticism, unless in actual practice acceleration factors operate during the actual life of a part. On the other hand, except perhaps in the case of accelerated corrosion tests where the results of chemical action may mask ambiguously what really does happen, accelerated tests do often give very valuable comparative results in the case of the mechanical behaviour of metals, or at least of some metals, under stress. In regard

to short-time fatigue tests, the difficulty, as H. F. Moore, of the Illinois Engineering Experiment Station—who has done such brilliant work on fatigue of metals—points out in Bulletin 205 recently issued (University of Illinois, price 20 cents), is that for the most part they seem to detect slip, rather than the starting of a fatigue crack, and there is at present no clear relation known between the beginning of slip, with consequent mechanical hysteresis, and the start of a fatigue crack. Moreover, ferrous and non-ferrous metals appear to behave differently in respect of the sequence of these two events.

#### The Ikeda Short-Time Test

THIS, notwithstanding the measurement of the change of electrical resistance under repeated stress, might be expected to throw light on what really happens, as the opening up of a fatigue crack might be assumed to affect the resistance of a piece of metal fully as much as would the phenomenon of slip without fracture. A method based on these considerations has been devised by S. Ikeda as a short-time test for endurance limit, and is described in the Bulletin under discussion. The disposition of the apparatus and the mode of applying the test is fully described. From its application to ferrous metals as diverse in composition as "Armco" iron, and tool steel, and to brass, monel metal and 99.98 copper, the following conclusions were drawn:—(1) the test gave results fairly close to those given by the ordinary long-time fatigue test (on the whole, the Ikeda test gave results, for endurance limit, slightly lower than those obtained by long-time tests, and are hence on the "safe" side); (2) it established that the total change in the electrical resistance of a metal subjected to repeated stress depends on change in temperature and change due to structural rearrangements within the metal, and (3) for ferrous metals the temperature effect appears to be the dominant one. The test appears an easy and inexpensive one to apply and one, moreover, which affords useful indications which can be obtained in quite a short time.

#### The Grusonwerk, 1855-1930

"KRUPP" Grusonwerk—two words—but in those two words how much is there revealed of Germany's industrial history and present day achievement!" So runs the opening sentence of the imposing brochure, which has been produced by Fried. Krupp Grusonwerk A.G. Magdeburg, Germany, to celebrate the 75th anniversary of the Grusonwerk. The evolution of the great German iron and steel industry in the middle of the last century and the tenacity of purpose which was the predominating feature in the characters of the men who controlled it forms a chapter without parallel in industry. How Gruson came to invent chilled iron armour is one of those questions which will probably never be decided, but the success of this material produced an amazing development in his new works built at Magdeburg-Buckau in 1869-71, and his methods were always to build and produce "as big as possible—as fast as possible." For a long time Krupp and Gruson produced side by side with a singular absence of friction, Krupp working on crucible steel and Gruson on cast iron, each developing his own material to an unsurpassed degree. Gradually, however, they entered each other's fields, their interests clashed in earnest, and fusion took place in 1892.

Afterwards the production of war material was more and more transferred to Essen, the Magdeburg establishment seeking its further development in the field of more normal industrial activities. During the world war, the production of war material again occupied the Grusonwerk, but on the cessation of hostilities it was completely dropped. Since then, production may be said to have been built up on two main pillars. One—the knowledge and experience in the manufacture of chilled iron inherited from Hermann Gruson; the other—the manufacture of high grade cast steel and special manganese steel, activities springing from that fructification effected by the union with Krupps.

A secondary story which runs through the sections treating of the specialities of Krupp Grusonwerk is the way the heavy German industries have faced and mastered the difficulties of post-war years. In the German potash industry, for example, as in many others, the war resulted in the relatively small number of works remaining having to increase their

production vastly and, consequently, to improve or extend their mechanical equipment. Grusonwerk energetically tackled the fresh problems and succeeded in evolving on a large scale crushing, screening and transporting machinery, which at the same time marked a definite advance over older plant.

#### Adhesion of Protective Coatings

IN the adherence of a protective coating to the metal it is intended to protect, the nature of the basis metal, and in particular of the surface of that metal, plays, as all electroplaters are aware, an exceedingly important, if sometimes somewhat obscure, part. Its function, naturally, influences the adhesion of coatings of all kinds: painted or sprayed; Parkerised; sherardised; coslettising; calorised, or any of the hundred and one methods currently practised. In homogeneous metals, such as rolled strip and others in which the nature of the surface does not differ essentially from the rest of the material, the problem is simplified, which is one of the reasons why "Armco" iron, for example, is such an admirable base for enamelling. In cast metals more difficulty occurs, as they usually possess a well-defined "skin." This is particularly the case in regard to cast iron, and a very interesting study of the blistering phenomena in the enamelling of cast iron is ascribed to this cause in one of the latest Research Papers issued by the Bureau of Standards of the United States Department of Commerce (No. 179).

#### Blistering of Enamel on Cast Iron

THE research, which was undertaken in response to a request by the American Ceramic Society, was conducted by A. L. Kryniwsky and W. N. Harrison, and attributes the blistering that occurs in the enamelling of cast iron to the action of the "microchill" which grey iron acquires in freezing and cooling. During the enamelling process the combined carbon of this skin tends to break down to a nascent and readily oxidisable form of carbon, which evolves CO and CO<sub>2</sub>, both of which gases were found in the resulting blisters. This suggested that the removal of the outer skin might provide a remedy, and by removing the surface layer by sand blasting, or "burning out," a practical means of avoiding blistering of sound castings was attained. The Report says that there are probably two kinds of non-blistering iron, one in which little combined carbon is present at the surface, and another in which it is stabilised. During the early stages of enamelling both blistering and non-blistering irons evolve gas, owing to the quick oxidation of submicroscopic graphite, which escapes before the enamel has fused to a retentive condition. Addition of graphitising agents such as silicon may be beneficial, but it is harder to prevent the formation of the microchill than of the ordinary, or macrochill; hence removal of the surface is the best method of securing a sound enamelled surface.

#### Swiss Aluminium Industry

FRESH capital for extensions and loan repayment is being sought by the Swiss Aluminium Industrial Co., Ltd., of Neuhausen, founded in 1889, for the purpose of utilising industrially electro-chemistry and metallurgy, with special reference to the production of aluminium and its alloys.

In 1928 some 18,000 tons of raw aluminium were manufactured, of which about 15,000 tons were exported in the raw state to foreign countries. In the same year Switzerland imported approximately 25,000 tons of copper and zinc. Thus Switzerland appears to import foreign raw materials for working up into finished products, while she exports five-sixths of the raw materials she herself produces.

Although Switzerland imports so much foreign raw material and uses in her own factories so little of her own home-produced raw material, the 3,000 tons of aluminium she does use is a great advance on former years, such as 1918, in which year she only used 5 per cent. of the total production for working up into finished aluminium goods. The value of the exported half-finished aluminium goods from Switzerland in 1929 accounted for 45 per cent. of the total export value, while as to quantity the aluminium goods made up 25 per cent. of the 1929 exports. New aluminium alloys which have been recently produced make it probable that Switzerland will be able to develop her activities in this line in the future.

## Trade, Commerce, Finance : The Month in Review

### From Our Market Correspondent

THE iron and steel trade continues to mark time through this dull season. It was not to be expected that the month of August would show any improvement; indeed, it would do well to keep up even to the low standard of the previous months, as the influence of the holidays, in addition to the terrific heat of the last week of the month, will have had a material effect on the amount of business done and on the production of steel in the works. The depression in the industry is as acute as ever, and there are as yet no definite signs of revival. The course of events during the past few weeks has been very discouraging. Holiday stoppages have been prolonged, blast furnaces and steel furnaces have been laid off, and production has steadily declined. The markets still lack confidence, buyers being in no hurry to make forward contracts, and the amount of current business which they can place is relatively small. The only consolation one can find is that we are about at the end of what is always the slackest period of the year, and it is, therefore, not unreasonable to hope for some improvement, however slight, in the coming month. There are no solid grounds for expecting any big change, but during the last few days there does appear to be a little more cheerful tone, and even that may be sufficient to start business moving more briskly. Most of the trade reports indicate the probability of an improvement as soon as the holidays are out of the way.

#### Need for Forward Buying

Buyers have held off the market for so long, encouraged as they have been by the low prices to wait for still further reductions, that they will soon be compelled to consider the renewal of contracts. The policy of buying in the open market has been pursued because of the low level of prices, and can be continued only so long as the depression lasts. The first sign of a trade revival will bring out the usual forward buying; and the liveliness of the past week may be the prelude to this movement. Whether it will result in any quick or extensive increase in working specifications is not so certain. Yet that is what is so urgently needed. Order books have been reduced to a state which gives no certainty of future operation, even for a week ahead, and the production programme in many of the mills has had to be reduced below the economic level of working. It is somewhat depressing to see the way in which the works scramble to secure any order which comes on the market. Delivery is promised not in weeks or even days, but in so many hours after the order is placed. In those sections of the market where prices are free, buyers are offered the double attraction of low quotations and immediate delivery; and some of the quotations reported are ridiculously low. Evidently some of the works consider it preferable to face a fairly heavy losing margin in the price than to reduce production still further. Truly, the steel makers are at present between the devil and the deep sea.

#### Hopes of Trade Revival

It is too early yet to gauge the real value of the activity which has been shown on the stock exchanges during the closing days of the month, although the position may be clearer by the time this article is in print. It may be due merely to professional buying to cover bear sales, or the exceptionally low level to which even the soundest industrial shares had fallen may have attracted buyers who wished to get in at the bottom; or the meaning of the movement may be that in the exchanges there is some kind of foreknowledge of a general revival in trade. At any rate, this little flutter has had a brightening effect, and encourages the hope that we may be out of the present depression sooner than was thought possible a month or two ago. Political rumours have been floating about, and if anything comes of the suggestion that has been put forward of a 10 per cent. revenue tariff on all imports other than raw materials, things will certainly get a move on. Such a proposal does not by any means go far enough to meet the special condition of the iron and steel industry, for which a protective tariff that will put it on a fighting level with foreign competitors is absolutely necessary, but it would be a move

along right lines and would open the way for a policy which would give the basic industries of this country their rightful chance of preserving their own home trade, which in turn would enable them to secure a fair share of the world's export trade. Slowly but surely pressure from both inside and outside seems to be forcing the present Government to take action in the direction of the abandonment of our free imports policy.

#### The Individualist Policy

Meanwhile the cry still goes up for an extension of the process of rationalisation, although the general belief in that as a panacea does not seem to be quite so strong. There is an important body of opinion amongst industrial leaders who favour the continuance of the individualist policy. The manner in which rationalisation has been carried out in this country makes one inclined to agree with them. There is still good scope here for the individual works where quality and service count for more than quantity and output, and given protection from unfair foreign dumping, such works will be able to hold their own with any of the combines that have recently been formed, and will play their full part in restoring the industry to something like its former healthy condition.

Quotations for semi-finished steel and for finished steel products from abroad are extremely low, although they are not attracting very much new business, simply because buyers on this side have not the business to place. Nevertheless, there is a steady inflow of steel which the British works would be very glad to have the opportunity of supplying. This applies particularly to the special free-cutting steel bars coming from Germany and Czechoslovakia, which is being sold at prices which the English makers cannot meet, and which is spoiling what was previously a reasonably remunerative product for our steel makers who produce this class of steel.

#### Pig Iron Prices

There have been further interesting movements in pig iron prices during the month. The reduction of 2s. 6d. per ton which was made in July by the blast-furnace owners in the Midlands was followed by another similar reduction early in August. This enabled the Midland makers again to encroach on the local trade of the Cleveland district, and, therefore, the Cleveland makers were forced to make a drastic cut of 4s. per ton in all their prices about the middle of the month. Although the effect of this reduction has not yet made itself fully known, it will probably check the outside competition. It should also stimulate buying as it is hardly likely that prices will go any lower. The hematite market has taken on a firm tone, as the reductions in output make it possible for makers to resist any further demand for lower prices.

The steel makers have had no cause for cheerfulness in their experience of the finished steel markets recently. The scarcity of orders has been a serious problem, and it will be interesting to see how far this is revealed in the production figures when officially published. Keen competition continues in small bars and boiler plates as well as in soft billets, but there has been so little business about that price cutting has not been of much avail. One satisfactory feature of the trade is the growing demand for steel arches and props for the collieries. In many districts these are altogether superseding the wooden supports, and there is quite good business to be done in them. At present the re-rollers supply a large proportion of the demand, but some of the steel works, also, are finding it a very useful line of business, and the direct supply from the works will probably increase.

There is no change in the official prices for steel plates and sections. If there should be any noticeable improvement in trade between now and the next meeting of the joint associations in October there may be then no alteration, although if the present depression continues a reduction is very probable.

The number of blast furnaces at work at the end of July showed a decrease of 28 since the beginning of the month. The output of pig iron fell to 486,100 tons, compared with 563,200 in June. The output of steel was 621,400 tons, compared with 600,100 tons in June.

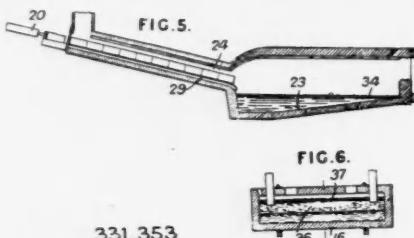
**Some Inventions of the Month****By Our Patents Correspondent***Abstracts of other Patents of metallurgical interest will be found in our Patent Literature published weekly in THE CHEMICAL AGE.***Alloys**

ACID-RESISTING iron alloys described in Specification 331,414, dated August 2, 1929, by K. M. Tigrerschiöld, of Fagersta, Sweden, contain 15-30 per cent. of nickel, up to 3 per cent. of chromium, 0.05-1.5 per cent. of carbon, 2.5-10 per cent. of silicon, and 0.2-2.5 per cent. of copper; the silicon may be wholly or partly replaced by molybdenum, and the nickel by cobalt. When molybdenum is present the content of molybdenum + silicon is preferably in excess of 10 per cent. Vanadium, titanium, tungsten, or manganese may be present in small amount.

ALLOYS, specially suitable for making sparking plug electrodes, comprise nickel, manganese, either chromium or silicon, and an ingredient which reduces the sparking voltage, such as an alkali or alkaline earth metal. The amount of chromium or silicon is preferably not above 10 per cent. Alloys are specified containing 88.94 per cent. of nickel, 1.5-2 per cent. of manganese, 4-10 per cent. of chromium, and 0.4-6 per cent. of barium. See Specification 330,401, bearing the International Convention date, April 1, 1929, by A. C. Spark Plug Co., of Flint, Michigan, U.S.A.

**Copper**

A PROCESS for melting and refining copper in a reverberatory furnace is described in Specification 331,353 (Jensen), a communication from American Metal Co., Ltd., of New York, U.S.A., dated June 7, 1929. Ingots of the metal, preheated to a temperature somewhat below the melting point, are plunged separately into a bath of metal in the melting furnace. They are



331,353

fed by a ram 20 down an inclined flue 24 into the deeper end of the bath on the melting hearth 23. To enable the hot gases to have free access around the ingots, the latter move on tracks 29 raised above the floor of the flue. The superheated metal is discharged through a taphole 34 at the shallow end of the bath into a tiltable refining chamber 16 in which it is covered by a layer 36 of deoxidising slag and a layer 37 of carbon. The slag is maintained in an active state either chemically by the carbon layer, or electrolytically.

A PROCESS of hardening copper articles described in Specification 330,408, dated June 17, 1929, by N. O. Hedman, N. R. Näslund, and J. D. Larson, of Alvsbyn, Sweden, comprises heating to approximately 600° C. followed by cooling in a bath consisting of a mixture of concentrated sulphuric acid, concentrated nitric acid, and 5-10 grammes per litre of potash. The sulphuric and nitric acids are preferably in the proportions of about 14 to 1 by volume.

**Extraction of Metals**

THE extraction of metals from their compounds is effected by causing hydrogen, or a mixture of steam and hydrogen, or steam and hydrogen in succession, to bubble through a molten and freely mobile bath consisting of or containing the compounds, according to Specification 329,159, dated February 26, 1929, by E. A. Ashcroft, of Ashburton, Devon, and F. E. Elmore, of Boxmoor, Herts. The reduction is specified of (1) lead chloride melted over molten lead at 800-850° C., (2) stannous chloride over molten tin at 600° C., (3) cuprous chloride at 500° C., and (4) iron chloride at a temperature between those required for tin and lead. The process is also applicable for obtaining silver, gold, platinum, cadmium, nickel and other metals, the temperature in each case being as nearly as possible the volatilisation temperature of the melt. Metals which are non-molten at the temperature of the molten compound are thus obtained in granular form. Reference has

been directed by the Comptroller to Specifications 7,353<sup>49</sup> and 193,475.

THE extraction of nickel, iron, copper, and other metals from sulphide ores containing nickel is effected by heating the crushed ore at about 700-1000° C. with excess of air to burn off the unwanted proportion of sulphur and to convert the iron into oxide or sulphate. An easily decomposable, chloride, e.g., sodium or magnesium chloride, is then added and the roasting is continued at a temperature not above 650° C. to ensure a complete formation of the chlorides of nickel, copper, silver, and palladium. These chlorides may then be removed by raising the temperature to volatilise them, whereupon they are condensed in water. The remaining charge is then reduced and the iron is separated magnetically, the residue being treated for the recovery of gold and platinum. Copper and nickel may be deposited from the chloride solution by electrolysis at current densities of 1.5-5 and 15-20 amperes per square foot respectively, the silver and palladium being deposited as a mud at the bottom of the cell. Alternatively, the chloride solution may be evaporated, the residue heated to form the metal oxides, and the latter immersed in dilute acid to convert the copper oxide into a salt and precipitate the nickel oxide. In some cases separation of the nickel and copper chlorides by volatilisation is not possible, and it is necessary to cool the chlorinated ore and leach it with water or weak solutions of chlorine or chlorides. The solution may then be treated with an alkali carbonate to precipitate basic nickel carbonate, and thereafter with a basic hydroxide to precipitate copper hydroxide. See Specification 328,696, dated February 8, 1929, by F. L. Duffield, of London.

**Commercial Intelligence***The following are taken from printed reports, but we cannot be responsible for any errors that may occur.***Mortgages and Charges**

[NOTE.—The Companies Consolidation Act of 1908 provides that every Mortgage or Charge, as described therein, shall be registered within 21 days after its creation, otherwise it shall be void against the liquidator and any creditor. The Act also provides that every Company shall, in making its Annual Summary, specify the total amount of debts due from the Company in respect of all Mortgages or Charges. The following Mortgages and Charges have been so registered. In each case the total debt, as specified in the last available Annual Summary, is also given—marked with an \*—followed by the date of the Summary, but such total may have been reduced.]

ALUMINIUM CORPORATION, LTD., London, S.W.—Registered July 19, £150,000 debentures and premium of 25 per cent.; general charge. \*£1,123,465. December 2, 1929.

ANGLO-SCOTTISH TIN CORPORATION, LTD., London, E.C. Registered July 10, £2,213 and £2,213 debentures, to Tanganyika Goldfields, Ltd., and Kagera (Uganda) Tinfields, Ltd., both 79, Mark Lane, E.C.; general charges (except the Koto Bahru property, F.M.S.). \*—. April 11, 1930.

BALDWINS, LTD., London, E.C., steel manufacturers. Registered August 18, to charges additionally securing £1,000,000 1st debenture stock and £1,206,850 A debenture stock; charged on certain shares. \*£3,793,969. April 18, 1930.

SIPUT TIN CO., LTD., London, E.C. Registered July 22, £40,000 debenture, to Guthrie and Co., Ltd., 5, Whittington Avenue, E.C., and Singapore; charged on lands, mining leases and rights, particularly Perak mining leases Nos. 1,049, 1,050 and 1,051 with land adjoining together with dredge plant, etc., also general charge. \*Nil. October 9, 1929.

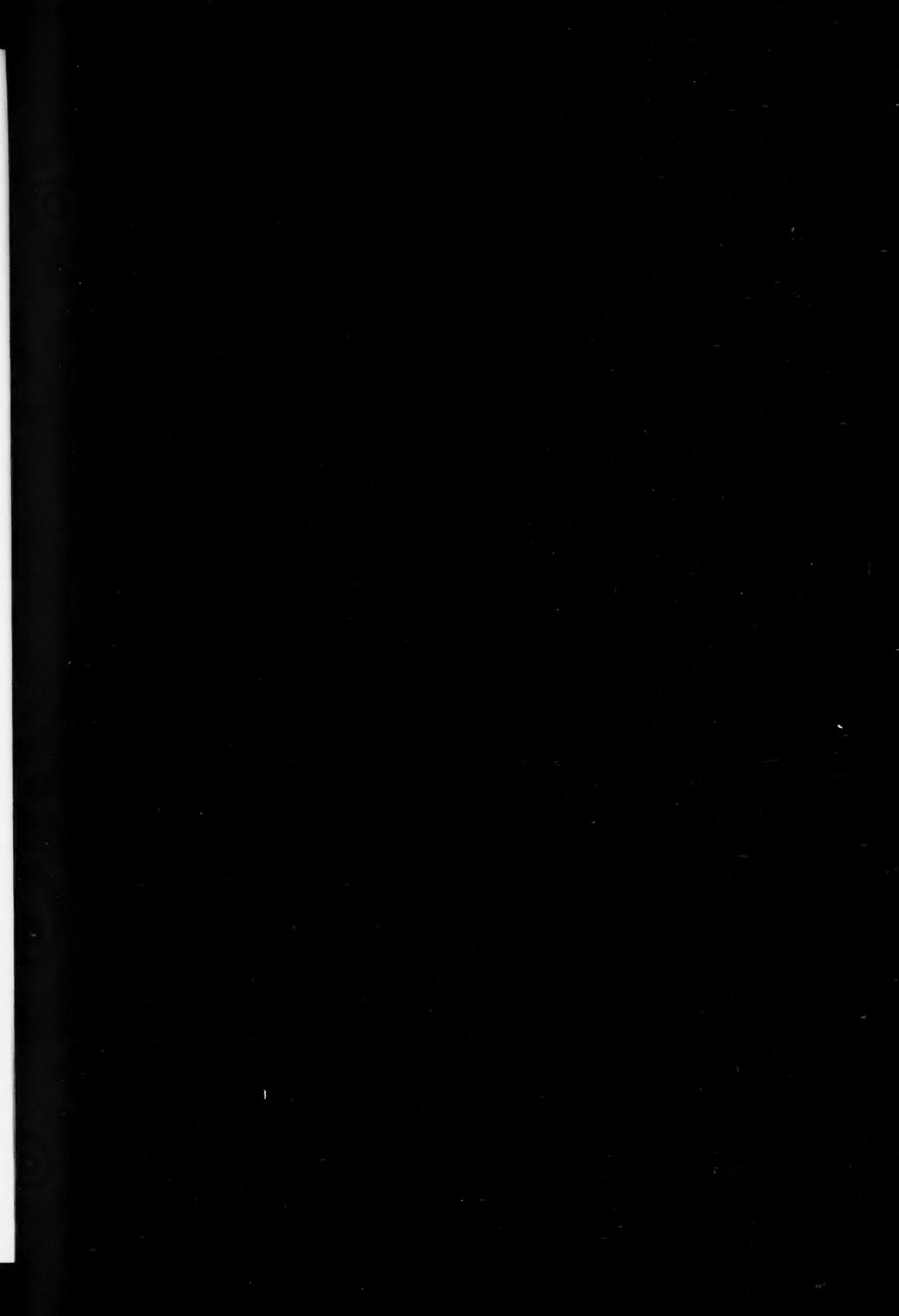
WIDNES FOUNDRY (1925), LTD. Registered June 10, mortgage and charge, to Bank; charged on land and foundry, shop and cottage property at Widnes, etc., also general charge. \*£60,000. January 14, 1929.

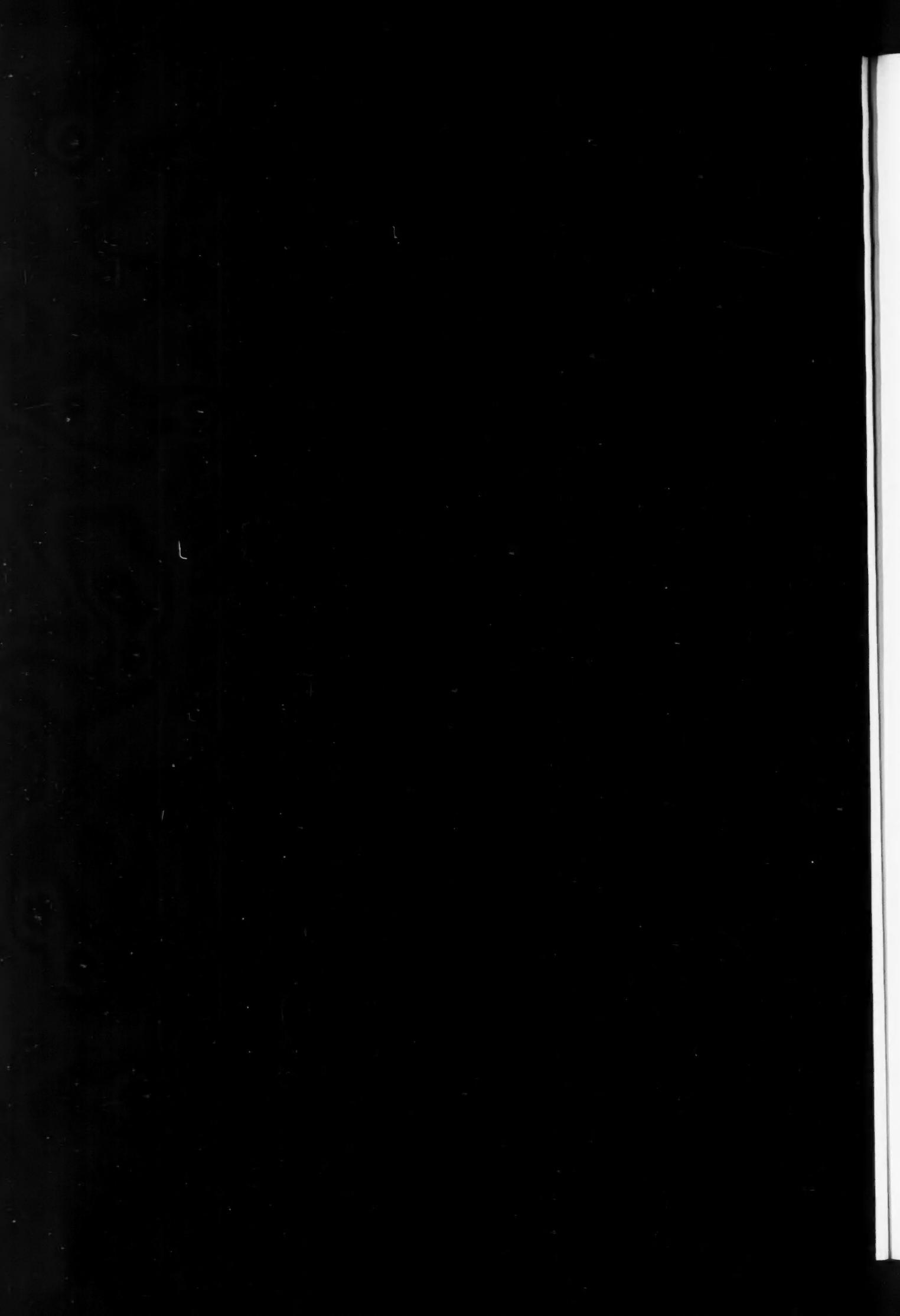
**Satisfactions**

BWANA M'KUBWA COPPER MINING CO., LTD., London, E.C. Satisfaction registered August 2, £950, part of amount registered April 13, 1928.

INTERNATIONAL ALUMINIUM CO., LTD., London, S.W. Satisfaction registered July 18, £5,600, registered July 26, 1927.

ROGERSON (JOHN) AND CO., LTD., Wolsingham, steel manufacturers, etc. Satisfaction registered August 7, £20,000, outstanding July 1, 1908.





# Monthly Metallurgical Section

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**NOTICE.**—Communications relating to editorial matter for our Monthly Metallurgical Section should be addressed to the Editor, THE CHEMICAL AGE, Bouvier House, 154, Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.

## High Frequency Steel Furnaces

By D. F. Campbell

A very informative paper on the wide field of utility for the high frequency steel furnace was read by Mr. D. F. Campbell, on September 16, at the autumn meeting of the Iron and Steel Institute in Czechoslovakia.

Below we reproduce some extracts from Mr. Campbell's paper.

THE melting of steel in an ironless induction furnace has been generally recognised as an efficient and economical method of making tool steel under conditions similar to those of the crucible process, but there is no justification for supposing that the induction furnace is limited to the simple metallurgical operation of melting small quantities of high-class raw materials to produce tool steel.

A survey of the present status of the ironless induction furnace will show that its field of utility is much wider than is generally supposed. As is usually the case with new developments, it is supplementing certain processes, replacing various types of furnace in part, and though its limitations of size will prevent its application to certain operations requiring large ingots or castings, the rapidity of operation will have an important bearing on steelworks practice outside the melting shop.

Ironless induction furnaces of the Ajax-Northrup type, having an hourly output of 20 to 25 cwt., have been operated since July 1929, and smaller units of 5 cwt. capacity have been producing steel regularly since October 1927, when the first tool steel from the ironless induction furnace was produced on a commercial scale in a Sheffield steelworks.

At present the largest units are furnaces of 20 to 25 cwt. capacity per heat, giving an output of 20 tons per day, but the design of furnaces of 3 to 5 tons presents no technical difficulties, as many details of construction are easier in furnaces of large dimensions, while large motor-generator sets can be built to operate at lower speeds, and their design is well within established practice. There appears to be no technical reason to prevent the construction of units of any size that is commercially practicable, though economic considerations indicate the probability that units of less than 15 to 25 tons will be adopted, as in the case of steel-making by the Bessemer or Thomas processes.

### Applications

At present, the steel-making operations to which these furnaces have been applied are as follows:—(a) The melting of high-class tool steels in acid-lined furnaces to replace the crucible process. (b) The melting of low-carbon steel and alloys. (c) The melting in an acid lining of rolling-mill scrap produced from steel made in basic-lined arc-furnaces. (d) The melting of heat-resisting steel and refractory chromium alloys. (e) The melting of nickel-chromium alloys. (f) The re-melting of manganese scrap. (g) The melting of hard materials such as tungsten carbide.

For making tool steel, 5 cwt. furnaces, melting one charge per hour, are commonly used. These are considerably cheaper to operate than gas or coal-fired crucible furnaces, and the labour is less costly and less difficult to obtain. In addition, certain qualities of steel containing complex mixtures of alloys can be made of a homogeneity unobtainable with the ordinary crucible furnace.

The most convenient time for making a heat of high-class steel is 1 hr. for 5-cwt. furnaces, and about 1½ to 2½ hr. in the case of 20 to 25-cwt. units. It does not seem to be advisable to work more quickly than this, owing to the time required to prepare moulds and cast ingots carefully. Faster melting is possible, but it is essential to allow a certain margin of time in high-class steel-making operations—a condition which does not apply so strictly in the case of mild steel for castings.

The electric furnace is used for melting steel and alloys requiring lower percentages of carbon than those obtainable by the crucible or arc-furnace processes—for example, magnetic alloys and extra-low carbon stainless iron. In the manufacture of magnetic alloys, a carbon content of less than 0.04 per cent. is necessary, and this composition is readily obtained by melting the highest grade of Swedish wrought iron with the necessary nickel and other alloys in high-frequency furnaces. The re-melting of low-carbon stainless alloys of the chromium and chromium-nickel series without any pick-up of carbon is a valuable use of the high-frequency furnace.

### Other Metallurgical Developments

In addition to the above established industrial uses, rapid progress is being made in determining the commercial value of the following metallurgical operations.

(1) The refining of steel is performed in basic-lined furnaces by slag reactions similar to those used in arc-furnaces. The refining operation is extremely rapid, owing to the circulation of the metal due to electrical forces. Furthermore, this movement constantly brings hot metal in contact with the underside of the slag layer, thus keeping it hot and maintaining ideal refining conditions at the slag-metal contact surface.

(2) Duplex processes—either with the open-hearth or with electric arc-furnaces in countries where the latter is the cheaper melting unit, where steel must be finished in an acid lining, and only basic steel is available—show considerable promise. For example, in certain Swedish works the following problem presents itself: It is proposed that a 1-ton high-frequency furnace should be installed. This furnace would be capable of making ingots large enough for 90 per cent. of the requirements of the shop. Occasionally, however, ingots up to 10 tons in weight would be wanted, and these would be of a specification requiring acid steel. It is accordingly suggested that a 1,000 kw. high-frequency equipment should be installed which would supply alternatively a 1-ton or a 10-ton furnace. The 1-ton furnace would be used for melting, and the 10-ton furnace for finishing steel previously melted in a basic open-hearth or basic electric arc-furnace.

(3) The conversion of molten pig iron into steel with the elimination of carbon, silicon, manganese, phosphorus, and sulphur, is carried out in basic-lined furnaces. The possibilities of converting liquid pig iron into steel are extremely interesting on account of the great rapidity with which the reactions occur owing to the intense circulation. The excessive over-oxidation of the basic Bessemer (Thomas) process is avoided, and by control of the circulation the conditions may be varied from those of a violently oxidising furnace to the comparative tranquility of an open-hearth or crucible furnace, which is desirable for finishing processes.

### Comparison with Open Hearth Furnaces

If a comparison be made between a steelworks equipped with six 75-ton open-hearth furnaces producing 6,000 tons a week, and ten 6-ton high-frequency units giving the same output and casting direct into ingot moulds, it will be found that the capital cost of the latter equipment will be considerably less, and the operating advantages would be numerous. Instead of 100-ton cranes and buildings of corresponding strength, the maximum lift in the melting shop would be limited to 10 tons, while a regular supply of ingots would be

available at regular intervals, which would avoid the necessity of large and expensive soaking pits and ladles. The fact that steel may be charged within a few minutes of starting work on Monday morning, and the possibility of completely shutting down furnaces during week-ends, are important features which are also attractive in the case of the Bessemer process. The space occupied by six 75-ton open-hearth furnaces, and ten 6-ton high-frequency furnaces, with two metal mixers in each case, would be about 70,000 and 32,000 sq. ft. respectively.

#### Recent Improvements

The present state of development justifies the statement that such refining furnaces can now be made. Details of metallurgical practice doubtless require further experience, and the difficulty of sample-passing requires consideration, but the accuracy of heat control in electric furnaces and the absence of combustion gases should give such uniformity of product as to reduce the necessity for sample-passing.

The possibility of such a steel-making plant producing a large output without the excessive capital expenditure which has characterised recent works, is certainly worth profound consideration.

Important improvements in equipment and process work have been accomplished in the last two years. The improvement in the efficiency of motor generator sets is remarkable. For 150 kw. generator sets efficiencies of 73 to 75 per cent. were acceptable two years ago, but now 83 to 85 per cent. is obtainable for this size of machine. This improvement

represents about 70 kw. hr. in normal practice, or about £200 per annum when using electricity at 6d. per kw. hr. and melting 100 tons per month.

The method of operating these furnaces is extremely simple; the method of charging involves the minimum of labour, and repairs to refractories are a small item owing to the small quantity used. The charging door consists of a hole in the working platform, and a 1-ton furnace can be charged in 1 to 2 min., if a reasonable quality of scrap is available. There are no electrodes, a constant cause of expense and worry for the furnace man, and the repairs to refractories between heats are almost negligible. It is customary to break off any accretions in the slag line, and sand the bottom if there is any excessive wear. Induction furnaces do not wear excessively at the slag line or bottom, but fairly evenly.

The following figures indicate the relative weights of the refractories in furnaces of the same daily output:

|                             |                      |
|-----------------------------|----------------------|
| Output : 20 tons per day.   |                      |
| (1) High-frequency, 20 cwt. | 4 cwt. of refractory |
| (2) Arc-furnace, 4 ton      | 260 "                |
| (3) Open-hearth, 8 ton      | 2000 "               |

The above figures indicate the great advantage of high-frequency furnaces for intermittent work, as the heating of refractories is a useless and expensive operation. The small weight of brickwork to be heated as compared with other furnaces results in low power consumption for intermittent work, and the possibility of pouring a heat of steel within 80 min. of starting with a cold furnace.

## Some Alloys Containing Silicon

### Extending Field of Application

*Silicon in an oxidised state is one of the most abundant elements, and in view of the considerable interest which has been taken in recent years in the utilisation of silicon in various branches of metallurgy, the following brief review of some alloys which have been found to be commercially useful is perhaps not out of place.*

THE most common and widely used alloy containing silicon is cast iron, which practically always contains some and often as much as 3 per cent., and even 4 per cent. Another class of material closely related to cast iron, the high silicon iron of the Tantiron type, usually contains about 12 to 15 per cent. silicon, and is used chiefly for chemical plant because of its resistance to corrosion by certain acids.

Silicon and iron form a series of alloys known commercially as ferro-silicons. These have a wide range of composition and contain from 20 to 90 per cent. silicon. These alloys are not often used as such in finished parts but are valuable as a means of adding silicon to other alloys, and particularly in steel making for deoxidation and manufacture of silicon steels. Ferro-silicon is sometimes used as a means of adding silicon to cast iron to produce high silicon iron castings. Large quantities of ferro-silicon are used annually in the steelworks of this country.

A development of outstanding importance was the discovery about 1900 by Sir R. Hadfield of the remarkable magnetic properties of silicon steels. The steel with about 4 per cent. silicon and the smallest possible amount of carbon and known under the trade name of Stalloy is extensively used for transformer sheets and telephone diaphragms. Its low hysteresis loss makes this material exceedingly valuable for electrical machinery. Mild steel with up to 5 per cent. silicon is now often used for castings and forgings. A silicon steel with 1.5 to 2 per cent. of silicon, together with a similar amount of manganese, is frequently used for springs under the name silico-manganese spring steel.

#### Alloys with Aluminium

The alloys of aluminium and silicon have of recent years come very much into prominence with the introduction of the 13 per cent. silicon alloy, and also the discovery of the process of modification of these alloys. They are fairly strong, having a maximum stress figure of about 13 tons per square inch when modified, but they are also light and can be made into die castings which have a relatively high strength to weight ratio. These modified aluminium-silicon alloys are now widely used for a variety of purposes, such as rail and road coach building,

frames of various kinds and general engineering parts. The alloy generally used in their manufacture is one containing about 40 to 50 per cent. silicon.

Small amounts of silicon are also found in other light alloys such as duralumin and Y-alloy, and some metallurgists believe that the silicon plays a part in the age-hardening of these alloys.

#### Alloys with Copper

Several useful alloys of copper which contain silicon as one of the alloying elements are now in use. The alloy generally used for making silicon additions is silicon copper, which has about 15 per cent. silicon. Copper alloys containing up to about 5 per cent. silicon are useful for corrosion resistance in certain media and are sometimes classified as silicon bronzes. Copper which has been deoxidised with small amounts of silicon is widely used for making electric wire. Only a very small amount of silicon is finally found in the wire, but its strength and electrical conductivity are sufficiently improved to warrant its use.

Another alloy which has been developed recently and is known as P.M.G. metal is a complex copper alloy containing some silicon and also other elements. It is a splendid alloy, and it is claimed that in several of its physical and chemical properties it is superior to Admiralty gun metal. Details of a new Japanese alloy termed Silzin Bronze recently published show that a copper alloy containing 10 to 15 per cent. zinc and 5 per cent. silicon is superior to gun metal for marine purposes.

#### Alloys with Manganese

The alloys of silicon and manganese most widely used are those employed in steel manufacture, namely, silico-manganese and silico-spiegel. The former contains about 65 per cent. manganese, and 25 per cent. silicon, the remainder being mostly iron.

The above does not claim to be a complete outline of all the classes of alloys containing silicon, but it serves as an illustration of the useful properties obtained by the judicious use of this element. Its field of application as an alloying element is very wide, and further development in the near future is to be expected.

## Metallurgical Topics: Monthly Notes and Comments

### From Our Own Correspondents

#### Two Important Events

THE past month has been a busy one for those who take their membership of the two leading metallurgical institutions seriously. There was just time for the more zealous to attend the business sessions of the Institute of Metals at Southampton on Wednesday and Thursday, September 10 and 11; take part in a few hurried visits after, and get back to London by Saturday in time to start with the Iron and Steel Institute party to Češko-Slovácko, to attend the autumn meeting of that body, at Prague, on Monday and Tuesday, September 15 and 16. Although few did so, there were some who did and they may be congratulated on having taken part in two very successful meetings. The Institute of Metals meeting really opened in accordance with a now established tradition, by the delivery, on the Tuesday, of the Autumn Lecture, by Professor Hanson, who took as his subject "The Use of Non-Ferrous Metals in the Aeronautical Industry." Professor Hanson is obviously a fervent believer in the future of the lighter alloys, and particularly in magnesium and its alloys. He regards it as a metal with a great future, and deplores the fact that this country is dependent on foreign sources for its supplies, the more so as its ores are plentiful within the Empire. Metallurgists who are acquainted with some of the recent developments in the manufacture and applications of this ultra light metal abroad will echo Professor Hanson's regrets, and it is indeed a mystery why its utilisation meets, in our own country, with so much apparent neglect.

#### Magnesium Alloys for Aircraft

THE best magnesium alloys compare, as Professor Hanson says, very favourably with aluminium aircraft alloys, both in the "as cast" and in the heat-treated conditions. One of them, AM7.4, is as strong, volume for volume, as heat-treated "Y" alloy, and definitely superior to the latter in specific tenacity. Sand cast alloys made with reasonable care are practically free from the pinholing which causes so much trouble in the case of aluminium alloys, and although they are not, as yet, easily die cast, the difficulties of applying that process to them are by no means insuperable. Magnesium, the crystal system of which belongs to the hexagonal group, is apt to be brittle unless great care is exercised in the working of it, and the working temperature is restricted to between about 420° C. and 450° C., but with slow pressure it extrudes admirably. As Professor Hanson points out, the corrosion effects of magnesium have been much exaggerated. The metal, indeed, forms a self-protecting film, like the oxide film on aluminium, and with reasonable care lasts as well as that metal.

#### Grain Refinement of Aluminium Alloys

OF the papers read at the autumn meeting, that by Dr. Rosenhain and Messrs. Grogan and Schofield on "Gas Removal and Grain Refinement of Aluminium Alloys," stimulated the best and liveliest discussion, not without some humorous elements to brighten it. The paper embodied the results of a number of experiments made at the National Physical Laboratory, in order to secure density and freedom from blowholes, in castings of aluminium and some of its alloys. The procedure finally adopted and advocated is to bubble titanium tetrachloride through the molten metal, and those who attended the annual inspection of the Laboratory last summer may remember seeing exhibited there sections of some small ingots which had been treated in that manner, and were remarkably sound and free from segregation. In the paper the authors describe titanium tetrachloride as causing the development of a very fine-grained structure, which they ascribe to the influence, not of the chloride, but of the titanium itself, and which persists after re-melting. "Y" alloy was the alloy chosen for the experiments, and it was found that the method produced high quality castings and further markedly improved the working properties of the material.

#### Fumes of the Tetrachloride

IN the ensuing discussion a certain amount of scepticism was evinced as to the practicability of the process in ordinary everyday foundry routine. Thus, Mr. George Mortimer,

while he had satisfactory results so far as the metal on which he had tried the process was concerned, had had trouble with the fumes, and seemed to anticipate further trouble on this score, with the Home Office authorities. On one occasion a little tetrachloride dropped on a damp patch of foundry sand, had not only "cleared the foundry," but very nearly cleared the district as well. He suggested soaking dried asbestos with the liquid, and placing it at the bottom of the melt. Dr. Rosenhain rather tartly described such a procedure as "a primitive works method," and as tartly disagreed with Dr. Haas, of Germany, who had suggested that the tetrachloride method would be difficult to apply, and that other chlorides would do as well.

#### The Harmet Process

THERE are seldom enough "scientific" members to secure a good discussion at foreign meetings of the Iron and Steel Institute, the "visits and excursions" providing the more serious side of the meetings. Hence what might have been a good discussion on casting large steel ingots under pressure fell a trifle flat, and although Dr. Hatfield spoke on the subject as chairman of the Committee on the Heterogeneity of Steel Ingots, he confessed to no first-hand knowledge of the process described in the very able paper by Dr. Kriz, of the celebrated Skoda works. The title of the paper was "The Heterogeneity of an Ingot Made by the Harmet Process." That process, practically in desuetude in Great Britain at present—we remember seeing it in operation at the Monk Bridge Iron Works some 18 years or so ago—is being used with admirable results in Skoda, not only on large ordinary steel ingots, but on nickel and other steel ingots weighing upwards of 40 tons apiece. The press employed exerts a pressure of over 11,600 lb. per sq. inch on the upper cylinder, and 735 lb. on the lower cylinder, which amounts altogether to about 3,800 tons. Compression takes from four to seven hours, according to the size of the ingot.

#### The Skoda Works

LATER on, when visiting the great Skoda Works at Pilsen, the members were able to see this process in application. At these works the manufacture of steel is purely subsidiary to the engineering departments, and the steel foundry is probably the largest in the world. In it castings up to 85 tons in weight can be made, while in the forge department it is possible to produce forgings in ordinary and in special steels up to 105 tons in weight. In this department the utmost care is taken in the heat-control and at the forges and presses, as well as at the reheating and annealing presses, all the latest heat recording and control apparatus is installed. Many of the visitors inspected the research department, which is one of the best equipped in the world, and were much struck by the extent to which all the branches of the work are correlated with the scientific investigations of the best methods and the selection of the best procurable materials.

#### Heat Economy at Czechoslovak Works

FROM a metallurgical point of view, however, the subsequent visits to the Trinec Works, and later on to those at Vitkovice, said by now to be the largest on the Continent, were revelations of what has been effected in the rationalisation of plant, equipment and processes, and the attainment of efficiency. At the Trinec plant an elaborate system is in vogue for the mixing of the blast furnace and coke oven gases in just those proportions found most suitable and thermally efficient for the various heat requirements of the different departments. The system is under automatic control, and by means of recorders, tell-tales and other devices, the least divergence from the composition of the mixed gases or their calorific value can be at once detected and rectified. Similar control is exercised at the Vitkovice works, and at both fuel economy has been brought to the highest state of efficiency and perfection.

#### Copper Recovery from Pyrites Ores

ALTHOUGH the method is neither novel nor confined to Trinec practice, the greatest interest was also evinced in the copper refinery plant at that works, where the roasted pyrites

extensively used in the blast-furnace charge is, after calcination, crushed, chlorinated-roasted, leached, and the solution passed in rotary precipitation chambers over scrap iron and the copper separated to the amount of over 1,000 tons of "cement" copper per annum. Zinc is also separated. Another feature which, at Vítkovice, excited great interest, was one of the only three plants in existence on the Loeffler high-temperature high-pressure steam system, yielding steam at 1,900 lb. per sq. inch pressure and 500° C. For the heat-resisting, non-creep parts of this plant it is understood that a special non-carbon-molybdenum alloy is employed, and the problem, which elsewhere has proved a baffling one, appears to have been solved satisfactorily, seeing that the plant has worked without interruptions for three years and given such encouraging results that a new and much larger one is about to be installed.

### Rare Metals in Glass

The physical properties—such as refractorability, absorption of heat rays and ultra-violet rays, transmission of luminous rays and reduction of glare—imparted by the rare metals to glass are dealt with in an article in the current issue of the *Vancoramic Review*, the organ of the Vanadium Corporation of America. Most of these metals give decided colours to the glass, but a few, such as cerium and vanadium, under proper conditions, produce colourless glass and, it is predicted, it will be these colourless glasses which will receive added attention in the future. Cerium nitrate is generally used for the addition of cerium to glass mixes and occasionally cerium borate and ceric oxide, the proportion of metal varying from 1.0 to 7.5 per cent. Cerium is of value in cutting off the ultra-violet rays; the glasses are very slightly coloured, and allow nearly all of the luminous rays to pass. The heat absorption is about 30 per cent.

Neodymium and praseodymium, when used in the proper combination, produce a neutral grey. By variation of the proportions the colour varies from a light ruby colour to greyish-blue and brown-green. They have a high absorption of the ultra-violet and infra-red rays.

Uranium in glass produces a brown to yellow colour with a greenish opalescence. The opacity for ultra-violet light increases as the glass is richer in metal. With about 4 per cent. uranium the glass is opaque to the indigo and violet down to the blue. The heat absorbed at most is about 55 per cent. The combination of uranium with nickel, chromium, cobalt or cerium, produces glass which cuts off practically all of the ultra-violet rays and with a proper proportion the glass is a neutral grey colour which prevents glare.

### Vanadium and Non-Shatterable Glass

VANADIUM has been the object of much investigation in connection with ultra-violet absorption. The faint colours imparted to the glass by practically all of the rare metals are objectionable when the glass is to be used as a protective covering for paintings as the true colour of the picture is obscured by the tint of the glass, producing an undesirable faded appearance. Although vanadium glass is practically colourless, it nevertheless cuts out practically everything below  $\lambda$  3580. Vanadium glass has the bright appearance and high transmission properties of ordinary glass. Vanadium finds additional use in glass used in the manufacture of non-shatterable glass, made by cementing two glass sheets to an intermediate sheet of celluloid. The ultra-violet rays passing through ordinary vanadium-free glass cause deterioration of the celluloid, which may be quite rapid where the glass is used on ships or automobiles in tropical or very sunny regions.

Titanium and zirconium, when used alone or in combination, produce desirable glass, and both give high melting glasses when used in excess of 20 per cent. of the mix. Zirconium silicate fused in the electric arc forms a glass which is practically infusible, melting above 2560° C. Apart from refractoriness, the important property of zirconia is its low rate of thermal expansion. Crucibles made from it can be plunged red-hot into water without damage. Zirconia allows practically all of the ultra-violet rays to pass, so that it is replacing fused quartz for this purpose.

Selenium is used as a decoloriser in conjunction with cobalt to remove the faint green tint present in most glass. The amount used is small as it has a very powerful action, one part being added for every 32,000 to 64,000 parts of sand.

Tungsten and molybdenum produce colourless glasses which show good ultra-violet and infra-red absorption. The light transmitted is practically 100 per cent. The introduction of the tungsten or molybdenum into glass offers difficulties, however, as the molybdenum salts are rather volatile and those of tungsten highly infusible. Further study should lead to an increasing use of these elements.

### Aluminium Brass Condenser Tubes

AN interesting development of work conducted originally by the British Non-Ferrous Metals Research Association has been the production of "Alumbro," the registered trade name of the aluminium brass produced by I.C.I. Metals, Ltd., a subsidiary company of Imperial Chemical Industries, and used in the manufacture of condenser tubes. It contains 76 per cent. copper, 22 per cent. zinc and 2 per cent. aluminium, and is the result of an improved method of casting which enables the metal to be poured into the mould without turbulence or splashing. The difficulty which had hitherto been experienced in obtaining sound castings was due to the fact that in the usual method of pouring splashing took place and the aluminium oxide film which formed on the surface of the molten metal became broken up, with the result that inclusions were formed, leading to flaws in the casting. Extensive laboratory tests revealed the fallacy of the belief that aluminium increased the tendency of brass to season-crack and other tests both by the corrosion Research Committee of the Institute of Metals and in actual practice showed that the resistance of brasses to corrosion was considerably improved by the addition of aluminium.

Repeated experiments with jet test apparatus showed 76/22/2 Aluminium brass condenser tubes to be extraordinarily immune from impingement attack, and under conditions in which ordinary 70/30 brass and Admiralty brass specimens almost perforated, the aluminium brass tube remained practically untouched. Further, by combining an electrical device with the jet test apparatus, it was shown that if the scale formed on an ordinary brass tube were scratched, corrosion could proceed. In the case of aluminium brass, however, the scale rapidly reformed and stopped corrosion.

Alumbro condenser tubes are the subject of a booklet, which has just been issued by the company and contains some remarkable records of corrosion-resisting properties of these tubes in actual service over considerable periods.

### Recovery of Indium

A PAPER by L. R. Westbrook, presented at the May meeting of the American Electrochemical Society, at St. Louis, dealt with the recovery by electrodeposition of the rare element, indium, which occurs in small amounts in process residues obtained in the purification of crude zinc liquors.

Indium is found in minute amounts in the majority of zinc blends, in some tin ores, pyrites, siderites and tungsten ores. It is a white, lustrous metal, soft and ductile, somewhat heavier than zinc, melting at 155° C., and boiling at about 1,450° C., and does not tarnish appreciably in air at ordinary temperatures.

In the purification of crude zinc liquors, indium is removed along with cadmium, lead and other elements and follows cadmium through the processing of the crude sponge, being later removed along with other impurities. After separation from other elements, the indium hydroxide press cake, obtained on final filtration, was dried and converted to oxide by calcination at 600–700° C., the resulting product being of high purity.

To recover metallic indium from this purified oxide, electrolytic baths of high concentration were used. The most satisfactory bath was made by dissolving 200 grams of  $In_2O_3$  in about 600 ml. of water containing 120 ml. of 96 per cent.  $H_2SO_4$ , adding 250 grams of  $Na_2C_6H_5O_7 \cdot 11H_2O$  (sodium citrate) and diluting to one litre. This bath was electrolysed at room temperature with a cathode current density of 2 amp./sq. dm. using platinum anodes and indium or sheet steel cathodes.

The molten metal casts easily into ingots, and glazed porcelain molds are quite satisfactory, but the surfaces should have a thin film of vaseline applied to prevent sticking. There is as yet no known use that would pay for the recovery of indium, and the amount known to be available is very limited.

## Trade, Commerce, Finance : The Month in Review

### From Our Market Correspondent

If any proof were needed of the growing depression in the iron and steel trade, it is to be found in the official returns of the outputs for the month of August. In comparing that month with the earlier months of the year, it has to be remembered that the holiday season always makes August the quietest month of all, and this will partly account for the decrease on July figures. But the fall in production when compared with August of last year is most marked. Here are the figures :—

|                    | Pig Iron.    | Steel.       |
|--------------------|--------------|--------------|
| August, 1929 ..... | 682,000 tons | 753,300 tons |
| August, 1930 ..... | 416,700 "    | 451,300 "    |

The reduction in the pig iron production follows on the damping down or blowing out of blast furnaces in all the iron making centres, necessitated by the ever dwindling demand for pig iron. On the North-East Coast, for instance, there are now only 25 furnaces in blast, whereas a year ago there were 44.

There is no market for basic iron. Those steel works which still have to buy their iron for steel making have either large stocks or are trying to work off old contracts; and the steel works with their own blast furnaces are reducing the output of these furnaces to the lowest possible level to avoid putting iron into stock. The steel makers are struggling hard to keep going, but from time to time reports appear of the closing down of one section or another. The plight of the South Wales steel works is well known. The Dowlais works of the Guest-Keen-Baldwins Co., which were only recently reopened, have again been put on day-to-day notice, owing to trade depression. On the North-East Coast, Dorman Long and Co. announce that their Acklam steelworks are to be stopped for the same reason. It will not be surprising to hear that other works are following suit, as those which are running are not on full time and it is with the greatest difficulty that sufficient orders are secured for even part time operation.

### Foreign Steel Prices

Now let us consider some other figures. A recent report stated that during the previous three months 40,000 tons of foreign steel had been imported into South Wales. Again, in a recent month 17,000 tons of small steel bars were imported from abroad into the Birmingham district. These bars are chiefly for the motor car industry and other special trades, all of which could be, and most of them used to be, supplied from the British steel works. Definite offers are now being made of foreign small bars at prices varying from £5 5s. to £5 15s. per ton delivered into the Midlands, while billets are offered at about £4 15s. delivered. These bar prices are about £2 10s. per ton below our prices, notwithstanding the fact that most of the home works are having to sell bars at under cost price. It is noteworthy also that these foreign prices for delivery here are about £2 5s. per ton less than the home prices in the country of origin. It is obviously impossible for our steel makers to meet such absurd prices; and no one who knows anything about the matter imagines that the difference is due to greater working efficiency on the Continent.

It is simply a case of dumping, intensified by the recent breakdown in the European Cartel, and worse than we have known it for many years past.

### Trade Revival Prediction

Among those directly connected with the industry there is no expectation of any improvement this year from industrial causes. No one can see any grounds on which to base the hope of a revival. Depression and unrest are world-wide and are crippling purchasing power both here and abroad. The President of the Board of Trade predicts that the commencement of trade recovery may be looked for in the spring of next year, as commodity prices have now touched bottom, and therefore there must soon be an upward movement, which, however, will be of a very gradual character. It is to be hoped that he may be right. Given a fair chance, British steel works should be able to hold their own.

### An American Suggestion

A good deal of publicity has been given to the views of Mr. Replogle, who has been over in this country from the United States of America. He has been investigating the condition of the iron and steel industry here. He describes our steel plants as obsolete in the majority of cases, and he suggests the construction of a modern self-contained plant costing £60,000,000 and capable of producing 3,000,000 tons of steel per year. Apart from the initial difficulty of obtaining anything like such a huge sum of money now or in the near future, it is questionable whether this huge modern plant would achieve the results claimed. It might effect a small reduction in production costs, but it would soon find itself in a position similar to that of the existing up-to-date plants, having to cut prices below the economic level in order to obtain sufficient work. This American idea of size does not quite fit in with our specialised industry. It is not new and greater steel works that we want. Many of our existing plants are quite as efficient as the larger American works, while the older obsolete ones are gradually going out of existence. The future of the British steel trade lies as much in quality as in quantity of production, and no one can beat us there.

In spite of what any of the American critics may say, the British steel makers are doing all that is possible to restore prosperity to the industry, and they will succeed when they are given a fair field.

### Market Conditions

There are no changes to report in market conditions during the past month. The demand for all classes of iron and steel remains very limited, and forward buying is not yet noticeable. In the pig iron market prices are unaltered. No further reductions are likely, as outputs have been reduced to such a level that there is no necessity to cut prices. Buyers, however, are not yet convinced that the time is ripe for buying forward, and day to day purchases are still the rule. Depression is still acute in all sections of the steel trade. Semi-finished steel has felt the effect of the Continental disorganisation, and prices have weakened considerably. Finished steel orders are as scarce as ever. The next joint meeting of the Steel Association, which is to be held in the middle of October, will be awaited with interest, as there is an expectation of an alteration in prices. The prices of controlled materials have remained unchanged throughout the year, and it is very doubtful whether there would be any increase in business if a reduction were now made.

### The Institute of Metals

#### Forthcoming Activities

A FULL programme for the winter session has just been issued by the Institute of Metals, and includes over 40 meetings, to be held in metallurgical centres throughout England, Scotland and Wales. The programmes of the various sections have been drawn up to meet the special needs of local industries.

Among the subjects dealt with are metals and alloys of the future, metallurgy of some of the rarer metals, magnesium alloy castings, gases in metals, the extraction of copper, chromium plating, the applications of nickel in industry, unsoundness in metals, wire drawing, and applications of copper to the building trade. Works visits are also announced, as well as practical demonstrations of metal working in the lecture hall. The President of the Institute, Dr. Richard Seligman, will read the opening paper before the London Section on "Some Non-Ferrous Metals in Chemical Engineering," and will also contribute, in Glasgow, to an "Aluminium Review" that has been arranged by the Scottish Section.

Next year's autumn meeting, it is announced, will take place in Zürich. In 1929 the Institute met in Düsseldorf, and for 1932 a visit of members is being arranged to Canada and the United States. Copies of the programme can be obtained on application to the Secretary, Mr. G. Shaw Scott, M.Sc., 36, Victoria Street, London, S.W.1.

**Some Inventions of the Month****By Our Patents Correspondent***Abstracts of other Patents of metallurgical interest will be found in our Patent Literature published weekly in THE CHEMICAL AGE.***Electrolytic Processes**

THE production of pure aluminium, beryllium, chromium, and other metals is effected by electrolysis of their molten halide salts at temperatures below a red heat using anodes which are either of tungsten or of another metal, graphite, glass, porcelain, or other material coated with tungsten. No corrosion of the anodes by the chlorine evolved occurs. See Specification 330,791 (Johnson), dated July 1, 1929, a communication from I.G. Farbenindustrie Akt.-Ges., of Frankfort-on-Main, Germany.

**Gas Producer for Treating Ores**

THE utilisation of an ash melting gas producer for carrying out various metallurgical operations is described in Specification 332,306, dated June 15, 1929, by H. J. F. Philipson, of St. Etienne, Loire, France. The fixed carbon is in the proportion of about 30 per cent. of the weight of the charge, the slag being formed by high ash content fuels or by addition of substances having a base of silica, alumina or lime; the ratio  $\text{SiO}_2/\text{CaO}$  in the slag is suitably 0.9. The blast temperature may be varied between  $400^\circ\text{C}$ . and  $800^\circ\text{C}$ . to accommodate variations in the composition of the charge. Low grade cast iron may be added for conversion into silicious cast iron free from sulphur. Iron ores, either formed into briquettes or mixed with the fuel, may be added, the proportion of iron being 50 per cent. lower than that in a blast furnace, and not more than 15 per cent. of the charge. Ores containing zinc, lead, lime, potash or soda may be added, and the oxides of the volatile metals collected; in the case of sulphide ores the sulphur is retained by the iron in the charge. Any copper or precious metals present are collected as a matte at the base of the producer.

**Iron**

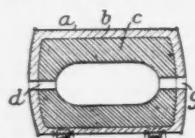
PIG iron having the properties of charcoal iron is obtained by melting down in a coke blast furnace scrap alone or admixed with ore and a flux mixture proportioned to produce a highly lime-basic slag rich in alumina. The slag should have an alumina content of at least 30 per cent. and its silicic acid content should be below 10 per cent. The desired alumina content may be obtained by addition of bauxite to the flux. See Specification 331,190, dated March 22, 1929, by M. Neumark, of Herrenwyk-Lübeck, Germany.

**Metallurgical Furnaces**

THE reduction in a high frequency furnace of a mixture comprising a metal compound and a thermo reducing agent is facilitated by incorporating in the mixture an electrical conductor or primer. The composition of the mixture may be varied according as a more or less energetic reaction is required. The charge is retained in the furnace until the slag has separated from the metal. Thus, for the reduction of a mixture of chromite ore and ferro-silicon, conductors of iron or iron-chromium are employed and may be in the form of filings, turnings, rods, sheets, grids, tubes, linings, plates, or rings, or may be residual metal from a previous heat. The ferro-silicon may serve as conductor if in suitable form and distributed throughout the charge. In the preparation of nickel-chrome alloy nickel-chrome conductors are used. The pots containing the charge may be heated to redness in a muffle before being placed in the furnace. See Specification 331,892, dated February 5, 1929, by W. B. Hamilton, of Birkdale, and T. A. Evans, of Whitefield, Lancashire.

**Melting Metals**

ZINC alloys and other easily fusible metals and alloys are melted by contact with the walls of a rotary furnace which has been heated up before the insertion of the charge. A suitable furnace comprises a drum *a* with an outer heat-insulating layer *b* and an inner refractory lining *c* of greater volume than the charge space. The firing and waste gas openings *d* and *g* are closed during the melting operation. The layer *b* is preferably of "Molera stone," a non-conducting material prepared from a porous



331,791

clay of Swedish origin. See Specification 331,791, bearing the International Convention date, August 20, 1929, by C. Brackelsberg, of Milspe, Westphalia, Germany.

**Porous Metals**

POOROUS metals, such as iron, nickel, cobalt, or mixtures thereof, specially suitable for electrodes for secondary batteries, are made by sintering the powdered metal at a comparatively low temperature, e.g.,  $500^\circ\text{--}700^\circ\text{C}$ . and thereafter subjecting the mass to one or more sinterings at higher temperatures, e.g.,  $800^\circ\text{--}1,200^\circ\text{C}$ . in a vacuum or in a reducing or inert atmosphere. Other powdered metals such as copper or reducible compounds such as oxides, hydroxides, or carbonates, may be added. See Specification 332,052 (Johnson), dated July 8, 1929, a communication from I.G. Farbenindustrie Akt.-Ges., of Frankfort-on-Main, Germany.

**Silicon Steel Sheets**

ACCORDING to Specification 331,511, dated April 2, 1929, by A. F. Murphy and W. Jones, of Zanesville, Ohio, U.S.A., silicon steel sheets for electrical purposes are treated by a process comprising pickling, cleaning away the acid, annealing, cold-rolling, and re-annealing. The sheets, flattened if necessary, are passed into a pickling bath containing sulphuric acid and a controlling agent such as that known under the trade name "Incontrol," stated to contain gelatinous material, blood, and preservatives. They are then washed, and the acid is neutralised with lime water. After scrubbing, the sheets are subjected to a spray of a suspension of air-slaked lime in water to form a coating which prevents their adhering together. The sheets are finally annealed, cold rolled, and re-annealed.

**Commercial Intelligence**

*The following are taken from printed reports, but we cannot be responsible for any errors that may occur.*

**Mortgages and Charges**

[NOTE.—The Companies Consolidation Act of 1908 provides that every Mortgage or Charge, as described therein, shall be registered within 21 days after its creation, otherwise it shall be void against the liquidator and any creditor. The Act also provides that every Company shall, in making its Annual Summary, specify the total amount of debt due from the Company in respect of all Mortgages or Charges. The following Mortgages and Charges have been so registered. In each case the total debt, as specified in the last available Annual Summary, is also given—marked with an \*—followed by the date of the Summary, but such total may have been reduced.]

BRITISH AND DOMINIONS FERALLOY, LTD., London, S.W.—Registered August 21, £800 secured notes, part of £15,000; general charge.

DOMINION STEEL CORPORATION, LTD., Sheffield.—Registered September 4, £5,000 debentures and bonus of 5 per cent. in certain events (secured by Trust Deed dated August 14, 1930), present issue £3,600; charged on part of building in Queens Road, Sheffield, also general charge, \*£1,000, September 14, 1929.

PRESSED STEEL CO. OF GREAT BRITAIN, LTD., Cowley (Oxon).—Registered August 26, £340,000 debentures inclusive of £200,000 already registered (secured by Trust Deed dated August 21, 1930, supplemental to Trust Deed dated March 21, 1927); charged on property at Cowley and Iffley and plant and machinery, etc. \*£200,000, May 27, 1930.

WHITEHEAD IRON AND STEEL CO., LTD., Tredegar.—Registered September 1, £50,000 debentures, part of amount already registered; general charge. \*£150,000, July 15, 1930.

**Satisfactions**

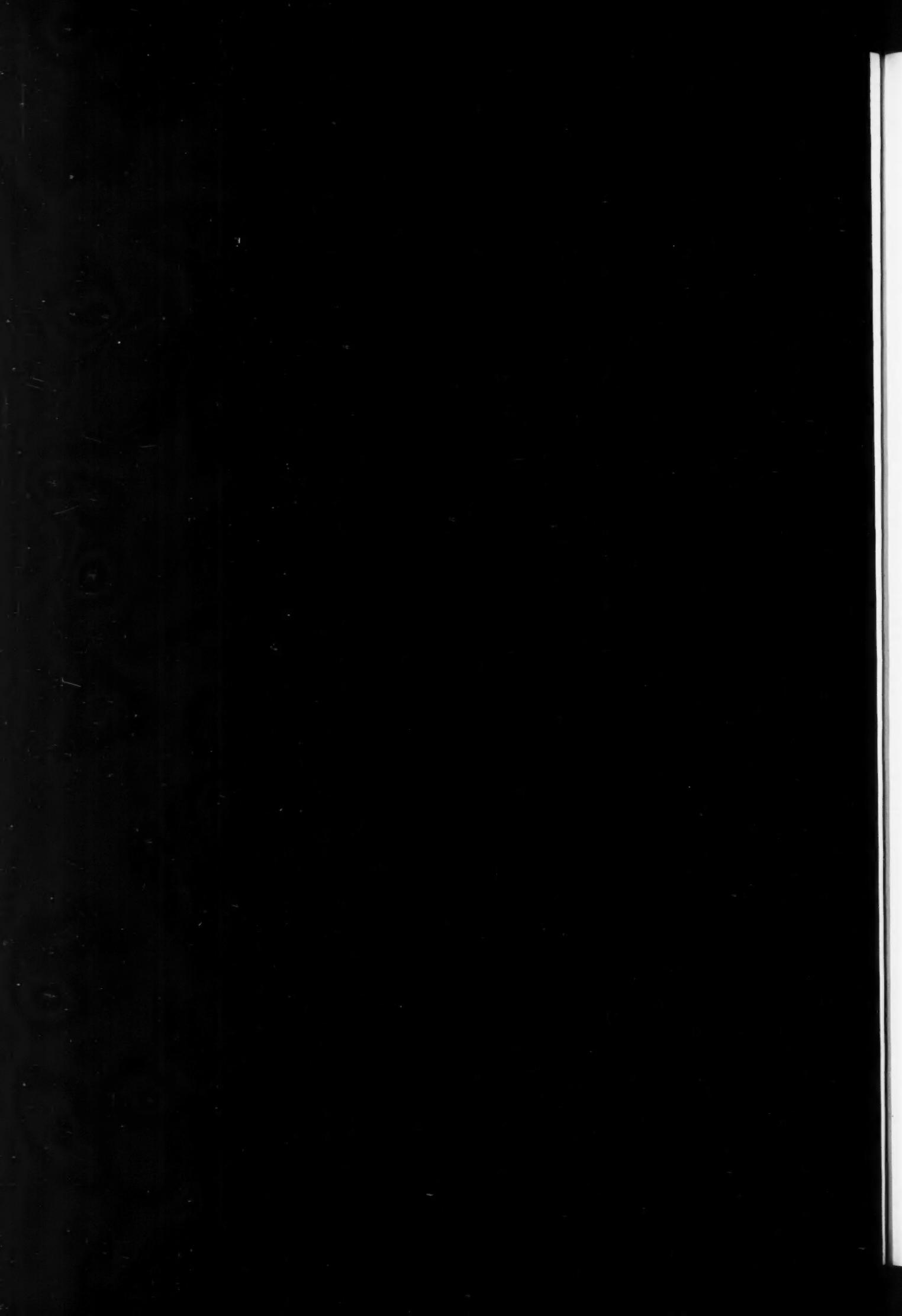
ALUMINIUM CORPORATION, LTD., London, S.W.—Satisfactions registered September 11, £250,000 registered July 3, August 7 and September 21, 1925, and £350,000, balance of £500,000, registered July 3, 1925.

BEARDMORE (WILLIAM) AND CO., LTD., London, S.W., steel manufacturers.—Satisfaction registered August 29, £111,783, part of amount registered January 10, 1930.

METAL AND IRON CORPORATION, LTD., London, E.C.—Satisfaction registered September 11, £626 12s. 1d. registered June 19, 1930.

UNITED STEEL COMPANIES, LTD., Sheffield.—Satisfaction registered September 5, £500,000, registered May 26, 1927.





# Monthly Metallurgical Section

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**NOTICE.**—Communications relating to editorial matter for our Monthly Metallurgical Section should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.

## Difficulties of the Electrodeposition of Chromium

By J. W. Cuthbertson, M.Sc.

The following extracts are taken from a paper entitled "Practical Difficulties in the Electrodeposition of Chromium," read by Mr. J. W. Cuthbertson before the Electroplaters' and Depositors Technical Society at the Northampton Institute, London, on Wednesday, October 15.

ALTHOUGH the electrodeposition of chromium has passed far beyond the experimental stage, the process is still, in some respects, unsatisfactory, and is beset by several practical difficulties. Provided that care is taken with the preparation of an article for "chroming," there will be less likelihood of trouble arising during this operation. Success depends, in no small measure, upon the skill and experience of the plater. When engaged upon repetition work the difficulties are not so great as those encountered in a shop which is undertaking work of all kinds.

It is impossible to lay too much stress upon the need for careful preparation of the work. Unless otherwise stated, it is assumed that the chromium is to be deposited on a nickel undercoating; this is not always necessary, but is generally advisable. Briefly, the work should be prepared on the following lines: After the usual grinding and polishing, the article should be thoroughly freed from grease; for this purpose the alkaline electrolytic cleaning vat is admirable. This is made up of caustic soda or potash contained in an iron tank, the latter acting as anode; cleaning is effected by passing a heavy current through the article, the violent evolution of hydrogen removing the grease from the surface. The article should then be dipped into a swilling tank, containing 10 per cent. sulphuric acid, and then rinsed in water. It should be scrubbed with either fine sand or pumice, the former for brass work, and the latter for steel, and again rinsed. On removing from the acid dip it is very important to see that no acid gets on to the scouring board used for cleaning with the sand or pumice. If this occurs, it will cause very serious trouble in the event of any cleaned iron or steel article coming into contact with it at a later date. It is essential that all traces of acid be removed from the surface of the article before it is placed in the nickel vat.

When a satisfactory finish has been obtained, the next stage is wiring and transference to the nickel vat. After cleaning and until nickelled, the work must not be touched with the bare hand, rubber gloves being used for the purpose. The extent to which handling will cause trouble will depend upon the individual, but in nearly all cases a greasy finger print will result in stripping or blistering of the deposit at a later time. This is especially to be avoided because the nickel deposit may appear to be perfectly sound, and it is not until an attempt is made to deposit chromium that a strip occurs; the whole process must then be repeated.

### Vat and Plating Solution

The type of vat used for chromium plating depends upon the work it is to handle. It is a great convenience and saving of time if small articles can be plated in a vat of medium size, and a larger one is to hand for heavier work. The size of a plant is limited by its cost, so that very often one vat has to deal with all the work. Even if only one dynamo is provided, capable of running each vat singly, the author is firmly of the opinion that better results are obtained if small articles are plated in a vat of small dimensions. Vats are often of insufficient depth. A deep vat is objected to on account of difficulty in operation, but if a platform is arranged around the vat at a height of 12 to 18 inches, no inconvenience should arise.

The results of a great number of experiments with different

solutions led to the conclusion that the best deposits are obtained from a solution of chromic acid and chromium sulphate in water. The tank is usually made of iron, the sides being glass lined, heated by gas burners from below. Iron is not an ideal material to use, but is as good as anything obtainable at a reasonable price. The anodes should be of lead. From time to time, attempts have been made to introduce iron anodes, but they are quite unsuited to the purpose. Unless electrolytic iron is used, it will dissolve in the solution, the rate at which this occurs depending on the carbon content. High carbon steel anodes are rapidly attacked. The greatest disadvantage in the use of iron or steel anodes is to be found in the effect upon the working of the bath. Iron tends to increase the concentration of trivalent chromium in the solution, and, as is well known, this leads to a decrease in the throwing power, and reduces the working range in which the vat will produce good, bright plate. Iron also increases the resistance of the solution. If lead anodes are used, a much lower value of the trivalent chromium content results, due to the higher oxygen overvoltage of these anodes. Intermittent plating, which, with iron anodes tends to raise the trivalent chromium in solution, has very little effect in this direction when using lead anodes. A certain amount of iron does dissolve from the tank, as is shown by the "ageing" of a new solution, but the quantity is usually so small that it has no serious effect. If, however, the iron increases appreciably, say to several grams per litre, the vat may be profoundly affected.

### Sulphate Ratio

The sulphate ratio of the vat is important. Generally speaking, we may say that there are four variable factors in chromium plating, viz., temperature, current density, chromic acid concentration and sulphate concentration. All these are intimately connected; thus if we have a bath of certain composition working at, say, 40° C., we may find that, with a current density of 75 amps. per sq. ft. a good deposit is obtained. If, however, we vary the chromium sulphate content without making any change in any of the other variables, we may obtain dull plate, or possibly nothing but hydrogen. In bright chrome plating baths, the ratio between chromic acid and chromium sulphate may be expressed in the form:—

$$\frac{\text{CrO}_3 \text{ molarity}}{\text{SO}_4 \text{ normality}} = \text{a factor, approximately 50.}$$

The exact value of this depends upon the composition of the remainder of the bath, and is generally higher, due to the presence of both trivalent chromium and iron.

When the right conditions are obtained, the chromic acid and the sulphate content of the vat should be determined, and an attempt made to keep to this composition continuously. The chromic acid is easily determined either by titration or by means of a hydrometer. The chromium sulphate estimation is not, unfortunately, so simple. If barium chloride is added to the plating solution, the barium sulphate thrown down is contaminated with barium chromate. It is, therefore, necessary to reduce the solution so that the chromic acid is converted into a chromic salt. There are several ways in which this can be done, hydroxylamine hydrochloride being very suitable for the purpose, although somewhat costly.

The temperature is usually kept at about  $40^{\circ}$  C. ( $104^{\circ}$  F.). This should be maintained as constantly as possible, the maximum variation permissible being  $\pm 3^{\circ}$  C. The current density should be adjusted with the same accuracy as the temperature. It is not difficult to estimate the approximate area of the surface of a piece of work, and, knowing this, the current is adjusted accordingly. The best results have been obtained with current density 65 to 85 amps. per sq. ft.

Whenever chromic acid is added to the vat, and this should be done daily if much work is passing through, it is essential to use only the pure material, free from sulphates. This is usually the only addition that is required. Some platers add chromium carbonate and chromium hydroxide to the solution. Such a practice is to be deprecated, as it can do nothing but harm by reason of increasing the trivalent chromium concentration.

#### *The Technique of Plating*

For the successful deposition of chromium it is frequently stated that the article on which the deposit is being made must gas freely in the vat. This is generally the case, but the gassing also causes a considerable amount of trouble. This is particularly noticeable in the case of articles which are perforated or have sharp angles and corners. Such articles require special preparation, all holes and apertures being stopped with corks. This is a tedious and troublesome process, and also necessitates having a large stock of corks on hand to meet all requirements. By exercising forethought and some ingenuity in the method of plating such articles, the need for corking can be minimised, although, in some cases, it will still be essential. Anode arrangement is also important in view of the poor throwing power of warm chromium plating baths.

The hangers used for conveying the current to the articles are made of copper rod, and a sufficient number of them should be used to carry the current required without overheating. They should make good contact with the cathode rods.

Large articles often present difficulties. We may consider the case of a car mudguard which was suspended in the vat by four hangers, two at either end, of ample dimensions to carry the heavy current necessary. When first attempts were made to deposit chromium on this in the manner described, the deposit was only sound in the region of the hangers, there was no chromium on the centre portion of the work, and some areas were badly blistered. By placing more hangers along the sides of the guard as well as at the ends, a good deposit was obtained. This brings out a point in connection with cathode current distribution. We do not know what this distribution is on a given article, and it is only through experience that an idea can be obtained as to its value, short of laboratory quantitative measurements.

If both sides of an article are to be plated, then holes must be corked. In the case of an article having a long open slot, this may be hung in the vat so that the slot is vertical. A miss will then occur at the upper end. If, however, we reverse the article at half time, the miss will now be at the other end, and both ends will be covered. In this case it is necessary to give the article a little longer in the vat than usual, as the two ends have had less deposit than the centre part. Trouble also arises at sharp angles, e.g., the corners of a rectangular frame. Such corners should be rounded off by corks.

#### *Small Articles*

Small articles are often not plated so well as larger ones. The reason is partly to be found in the prices paid for large and small work respectively. The remuneration obtained for the small articles often does not warrant the same amount of trouble being taken with them as in the case of a larger piece of work. The plating of small articles is often simplified by avoiding corking, and obtaining a complete covering of chromium by striking at a higher voltage than usual. The article is then kept at this voltage until it is on the point of burning, and the current then reduced to the normal figure. When removed from the vat, such a job appears to be satisfactory, but it gives a poor service. This is because there is so little chromium in the region of any holes; as soon as the working voltage is lowered to the normal amount, no more chromium is deposited on these areas, so that only the

uninterrupted surfaces of the article receive their full complement of chromium. A similar result is obtained if the solution is not working under optimum conditions. If the throwing power is not at its maximum, we may still obtain an apparently good deposit of plate, but this will be very thin in those areas where the current density is low. Hence the importance of keeping the vat in good condition at all times.

Barrel plating vats for taking small objects in large quantities do not give very good results. In one type investigated by the author, the deposit obtained on small screws seemed to be satisfactory, but was of a bad colour. Screws should all be separately wired.

It is sometimes necessary to examine the progress of an article in the vat, and this necessitates removal from the solution. This should be avoided whenever possible. If it is essential, the article must be thoroughly rinsed before returning to the vat, and also dipped into hot water to bring it up to the same temperature of the solution. If this is not done a bad greyish deposit will result.

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#### *Effect of Base Metal*

The ease or difficulty with which a chromium deposit can be obtained on an article depends largely upon the metal of which this is made. Chromium can be deposited directly on copper, and this is often done in practice. One firm is producing car bumper bars of spring steel coppered and chromium plated without any undercoating of nickel. Such a deposit of chromium is softer than usual, and not so blue in colour. Nevertheless, the author can say from experience that bumpers so prepared give excellent results, as good as, and possibly better than, when nickel is used as the undercoat.

The best way of plating bumper bars and high carbon steel articles in general, is to acid etch them after they have been prepared in the usual manner. This is done in a lead-lined tank, containing 95 per cent. sulphuric acid, the article being the cathode. A very heavy current is used which rapidly falls off to zero when the article is removed to a swilling tank. The latter should be agitated by blowing in air, as only in this way is it possible completely to remove all traces of acid from the surface. The acid etch should always be kept up to strength. The article is then plated in the nickel bath prior to chromium plating.

Zinc bath die castings are often far from simple to plate. A good result is ensured if attention is given to one or two details. Firstly, it is better to nickel such articles in a separate vat made up rather differently from the vat used for brass and steel work. A single salt solution containing citric acid as the buffering medium will give a good deposit. The articles should not be left too long in the nickel vat. There is no necessity to try and deposit 0.001 in. of nickel in this case, all that is required being a good covering free from pinholes. If too thick a deposit is put on, it will often lift in the course of chromium plating.

As soon as a satisfactory nickel deposit has been obtained, the article should be transferred without delay to the chromium vat. It is also very important to nickel these die castings immediately after cleaning and polishing. If they are left lying about in the plating shop, even for an hour, the surface becomes tarnished, and the deposit may strip in chromium plating. This is to be avoided on account of the trouble entailed in stripping the chromium and nickel from the zinc alloy.

The effect of moving the article during chromium plating was tried in a number of cases, but without any very noticeable improvement. There is a plant now on the market in which the work travels around a circular vat between inner and outer rows of anodes. It is claimed that, in this way, greater uniformity of plating is obtained together with an increase in electrolytic diffusion. The design is commendable in some respects, but has the drawback that, for plating large work, considerable floor space is required.

## Metallurgical Topics: Monthly Notes and Comments

### From Our Own Correspondents

#### British Cast Iron Research Association

THE ninth annual report presented at the meeting of the British Cast Iron Research Association, held in London on Wednesday last, is a very creditable document, and the chairman, Mr. H. B. Weeks, and the director, Mr. J. G. Pearce, are to be congratulated on the work accomplished during the twelve months. Further investigations have been carried out on moulding sands and refractories, calculated to save the industry large sums annually if applied scientifically in the foundry, and work has been done in developing high silicon heat resisting cast irons, suitable for use as melting pots, annealing pans and boxes and retorts. To the high silicon iron, aluminium, and/or chromium may be added. The resulting material has been patented, and the trade name Silal adopted for it, registered.

#### The Balanced Type Cupola

AMONGST other investigations undertaken by the Association and referred to in the report is one on cupola melting practice. A new type of cupola embodying modifications based on experiments carried out by Mr. George Pate, is described. The principle involved is to have a set of main tuyères controllable by screw valves, so that the air from the wind belt can be throttled independently at each tuyère, while it can, at the same time, be diverted to two or more rows of smaller tuyères higher up in the wind belt, thus adjusting the wind supply to whatever portion of the melting zone appears to need special treatment. Since the closing of the main tuyères results in more air passing through the upper tuyères, the cupola is of a balanced type and its use will, it is confidently expected, lead to coke economies estimated to amount to at least £100,000 per annum.

#### On Getting and Keeping Up to date

MR. W. T. GRIFFITHS, the devoted hon. secretary of the London Section of the Institute of Secretaries for several years past has vacated his office on becoming chairman, and at the opening meeting of the session, delivered an address on "Getting Up to Date and Keeping Up to Date," in respect of the acquisition, filing and recording of technical information. Those who have occasion to discharge those dual tasks will appreciate how difficult they have become, and, as a matter of fact, the subject elicited an animated discussion. One of the speakers made a useful contribution to the solution of the problem involved by the suggestion that the highly specialised card indexes in the possession of many investigators, might be placed at the disposal of those interested in the subjects concerned. A general feeling was expressed that the various excellent abstracts of current literature published by many of the scientific and technical institutions, appear at intervals too long to serve the purpose of keeping their readers fully abreast of the latest information available. It may be remembered that Dr. Seligman, in his presidential address to the Institute of Metals, took the same view, and endorsed the suggestion made some time ago by Professor Hanson that it would be advantageous if the abstracts section of the Journal of the Institute of Metals could be circulated at more frequent intervals than in the six-monthly issues at present published. It is understood that a scheme of this nature is, as a matter of fact, under contemplation by the Council of the Institute. It is to be hoped that it will be found feasible to give it effect, perhaps in connection with the scheme for international collaboration in the collection and dissemination of metallurgical and metallographical information adumbrated some time ago by Dr. Rosenhain.

#### Steel in Building Construction

IT is well to know that concerted action is contemplated in regard to the removal of some at least of the many restrictions which impede the widest possible application of steel in building construction. At a meeting held on October 16, at the Institution of Civil Engineers, a resolution in favour of having standard practice for the employment of structural steel was unanimously passed by those present, who, in addition to all the more important societies and institutions concerned with engineering, building construction and architecture,

comprised representatives of the Government, of the Dominions and of the London County Council and twenty-six of the leading city corporations and municipalities in the country. Several statements prepared for the occasion were supplied, one by the Ministry of Health, upon which has fallen the mantle of the former Local Government Board, explanatory of the confused nature of the law, and the complex local Acts, regulations and by-laws operating "in restriction" of the use of steel; one by the British Steelwork Association, the body convening the meeting; one by the Steel Structures Research Association, which is one of the branch bodies under the Department of Scientific and Industrial Research and one, finally, by the Engineering Standards Association. The importance of the occasion may be illustrated by the two following considerations: (1) that there is a lack of precise knowledge of the strength characteristics of steel frame buildings under load, and the technique of stress calculations in this branch of engineering; and (2) that many structural engineers are dissatisfied with the range of sections available to them, most of them designed at a time when neither the material nor the nature of stresses was fully understood.

#### Correlation of Existing Stress Data

IT may be remarked that the metallurgical institutions themselves were not parties to the meeting, although the questions involved are as much metallurgical matters as they are the concern of the engineer. It is metallurgical progress which has rendered many new steel materials available, such as the high elastic limit steels; the nickel steels; and steels of the non-rusting kind, all of which are to-day available for building construction. It is on the metallurgist, again, that the task of securing soundness in steel devolves. The strength of materials depends as much, or more, upon their freedom from defects, and their homogeneity, as it does on the intrinsic properties of the particular steels, or other alloys used. Finally, it is the job of a steel maker to roll the sections required, and to schedule the mill operations and design the rolls accordingly. Still, although the task undertaken by the Steel Structures Research Committee appears to be confined to one aspect only of the problem of becoming up to date in the uses of steel, the co-ordination of much of the existing data, and of reports such as those which have been issued by, for example, the Institution of Structural Engineers, on Loads and Stresses, and on Steel Work Connections, together with the necessary recalculations of stresses in the light of the much greater knowledge now possessed, should greatly help manufacturers and users of steel to so economise and develop its applications that, by a not uncommon industrial paradox, more, rather than less steel will be used.

#### The R101 Inquiry

WHILE it is of course quite impossible to predict the results of the inquiry into the loss of the R101, which began on Tuesday last, there is little doubt that much attention will be focussed on the subject of the metals used in its construction, their strength, and whether, in the case of structures of this kind, the margin of safety is all that can be desired. The metals were, for the most part steel, duralumin, and "stainless," and all of these have been the subject of the closest possible study, under stresses of the most diverse kind. Those likely to be encountered in aeroplane construction are very well known, and the apportionment of metal to ensure adequately high specific tenacity well understood. For highly stressed parts high tensile steel, or nickel-chromium steel hardened and tempered, is, on a weight-strength basis, the lightest metal, but the criterion of the best metal in tension is by no means always the criterion of the best behaviour of thin rods, sheets, etc., under shear, or compression stresses. Again, the margin of safety in respect of a number of conditions is not necessarily sufficient in all, and while much has to be sacrificed in aircraft construction to lightness, there is authority for the statement that, in respect of the fatigue limit allowances made by many modern engineers in aeroplane design and construction, there is sometimes no factor of safety at all.

### Molybdenum in Steel

THE functions of molybdenum as an alloy in steel have received a considerable amount of attention in recent years, and the consumption of steels containing a small percentage of the alloy has grown at an amazing rate. In 1918 the annual consumption was only 25,000 tons, in 1925 the figure had increased to 150,000 tons, in 1928 to 400,000 tons, and in 1929 to over 600,000 tons. The effects of molybdenum in steel are to exert a powerful influence in improving the physical properties, in promoting fine grain structure, in eliminating temper brittleness, in reducing the effect of mass, and in giving permanency to the physical properties which enable the steel to resist abnormal conditions during service, and when subjected to wide temperature variations.

The whole subject of the recent history and functions of molybdenum in steel and iron is dealt with in an admirable pamphlet which has just been issued by High Speed Steel Alloys, Ltd., of Widnes. It sets out the uses and potentialities of molybdenum steels with statistics provided by recent research, and at the same time makes it clear that molybdenum is not a panacea for all the troubles of steel. Its merits, however, are very considerable, and it furnishes qualities which, in some cases, cannot be obtained in any other way.

### Effect on Physical Properties

MOLYBDENUM added to straight carbon steels greatly improves their physical qualities, but it is used chiefly in combination with one or more of the other alloying elements, nickel, chromium, vanadium, etc., when it has the effect of intensifying the beneficial properties of these elements at the same time as exercising its own special functions. The intense activity of molybdenum as an alloy ingredient of steel is shown by the fact that the maximum quantity added to steel other than high-speed tool steel, is 1 per cent., and that for most purposes less than one-half of one per cent. gives the desired results.

Elaborate tests show that the inclusion of small percentages of molybdenum—that is, from 0.1 to 0.6 per cent.—effectively overcomes temper brittleness, and has a pronounced beneficial effect in reducing the "effect of mass." The molybdenum acts by promoting depth hardening, and even with small percentages (0.10 per cent.) the "effect of mass" is diminished, whilst with percentages up to 0.60 per cent. practically uniform depth hardening is obtained on masses up to 8 in. diameter. The published tests show that nickel chromium molybdenum steel of a section 4 $\frac{1}{2}$  in. varied only by 6 Brinell numbers from outer to inner surface, and a section 11 $\frac{1}{2}$  in. varied by only 10 Brinell numbers from outer to inner surface. These were comparative with a variation of 15 Brinell numbers in a section of 3 $\frac{1}{2}$  in., and 51 Brinell numbers in a section of 7 $\frac{1}{2}$  in. of nickel steel not containing molybdenum.

Many chemical gas and boiler plants which operate continuously at high temperatures, and at the same time are subject to heavy pressures, call for steel having a high yield point which does not fall off rapidly as the temperature rises, and an impact value which also remains consistently high under the same conditions. A heat-treated nickel chromium molybdenum steel shows the high yield point retained at elevated temperatures; thus at 450° C.—a by no means uncommon temperature for working high pressure and temperature plants—a yield point of 34 tons per square inch is still maintained.

The addition of small quantities of copper and molybdenum to steel has a remarkable effect in improving its corrosion-resisting properties as well as other physical qualities, and provides a useful material for boiler tubes. The use of molybdenum as an alloying element in iron castings is another field that has been developed recently.

### Rust Preventive Agents

TESTS have recently been carried out on two new rust preventive agents, called herolith and tornesit, by the German Mannesmann Tube Works. Herolith is a synthetic resin, and is applied by brushing, prior to which scale is removed by pickling. Tubes coated with it have successfully withstood thorough mechanical and chemical tests, the former comprising tests for resistance to shock, scratching and conveyance by train or motor-lorry. Thirty-five corrosive agents were used

in the chemical tests, only three of which (hydrofluoric acid, caustic potash solution, and caustic soda solution) had any effect on the tubes. Dry heat up to 220° C. and intermittent temperatures up to 300° C. do not injure the coating. Steam tests, not yet concluded, have so far given good results. The other agent, tornesit, is a rubber product similar in appearance to cellulose. It can be brushed on the part to be protected without preheating being necessary, and dries in three minutes. It hardens to such an extent after a few hours that even heavy hammer blows do not damage the coating. In contrast to herolith, tornesit is resistant to alkalis, and is only attacked by hydrogen sulphide, water, benzol, acetone and aniline. It can be employed as a protective coating for iron and steel, wood, brickwork and concrete.

### Cylinders for Storage of Gases

THE British Engineering Standards Association have just issued a British Standard Specification for high carbon steel cylinders for the storage and transport of "Permanent Gases." This specification, which is based on the recommendations for ordinary commercial cylinders for the transport of the so-called "permanent gases," included in the First Report of the Gas Cylinders Research Committee of the Department of Scientific and Industrial Research, provides for high carbon steel gas cylinders for the storage and transport of such gases as atmospheric air, oxygen, nitrogen, hydrogen, etc., which, at the usual working temperature and the maximum working pressure of 120 atmospheres, remain in a gaseous state in the cylinder.

The specification includes particulars of tensile, impact, flattening and hydraulic stretch and pressure tests, together with a formula for the determination of the minimum thickness of cylinder walls. This specification is the first to be published of three specifications which are in the course of preparation by the Gas Cylinders Committee of the Association for gas cylinders. It is expected that the two remaining specifications, for low carbon steel cylinders for the storage and transport of "permanent gases," and for steel cylinders for the storage and transport of "liquefiable" gases, will be available shortly.

Copies of this Specification No. 399-1930 can be obtained from the British Engineering Standards Association, Publications Department, 28, Victoria Street, London, S.W.1, price 2s. 2d. post free.

### The Literature of Iron Alloys

A GREAT programme of investigation of world progress in the field of iron alloys is being undertaken in the United States, under the leadership of the Engineering Foundation and with the co-operation of over sixty American industrial and scientific bodies producing or using iron and steel. A fund of some £60,000 has been raised, and as a commencement of the programme, which will take five years to complete, a critical review is being conducted of all available literature in English and other languages. A list has been made of approximately 2,000 scientific and trade journals in ten languages, and believed to be complete for all periodicals which have been published for any period since 1890 in twenty-five countries. At the moment this literature is being searched for information on thirty-nine elements and compounds in twenty-three separate classifications, making a total of more than 800 classifications.

The review of the matter dealing with the combinations of iron and silicon is now nearly completed, a bibliography has been prepared, some laboratory research done, and a monograph drafted. Considerable work has also been done on the combination of iron and manganese, and these two projects are stated to have proved fruitful not only in information on their subjects, but also as a guide to the committee in devising methods for the whole enterprise. Through a form designed to expedite abstracting, all the information gathered on each one of hundreds of subjects can be readily assembled. As the review of literature progresses there is being built up a valuable body of reference material on which may be based later a service of great convenience to persons preparing programmes for research, patent claims, papers for technical societies and for other purposes. More than 3,000 abstracts have already been made and filed.

## Trade, Commerce, Finance : The Month in Review

### From Our Market Correspondent

THE complete absence of any revival in the iron and steel trade is not surprising. At the beginning of the month every indication pointed to a continuance of the slowly deepening depression which set in in the early part of the year, and this has been confirmed by the experience of the past month. Not only that, but November is likely to be just as bad. There are still no grounds visible on which to base the hope of an improvement, and the year will probably drag on to its close with the record of having been one of the worst in the history of the trade. We have to draw what consolation we can out of the thought, to which expression is being given in various quarters, that there may be a better outlook at the beginning of next year. There is, however, not much enthusiasm about it; hope has been deferred so long that there is little or no confidence left, and with the failure to secure response from the Government to the demands of the industry, the steel makers are growing decidedly pessimistic.

It is all very well for ministers to talk, it is urged, about the slowness of the heavy industries in not making more use of their opportunities, and to threaten Government action to compel the wholesale adoption of rationalisation schemes. These are not going to save the situation, if the record of what has already been done by rationalisation is a criterion. They have certainly put a premium on unfair competition. The procedure seems to be to carry on a campaign of senseless price cutting, then, when financial difficulties have become too great to be surmounted, to adopt some scheme of rationalisation which cancels the greater part of the indebtedness, if not the whole, and start again the process by cutting out competitors. The banks have rather encouraged this kind of thing, on more than one occasion, by forgoing the interest on overdrafts for a period. But no help or relief is available to those undertakings which have consistently striven to run on right lines, technically and financially. If the Government really wishes to help the industry, let it start by putting a stop to the wholesale dumping of foreign steel into this country. That is the only legal action that is wanted at present, not interference in the domestic management of the steel works.

#### Steel Association's Meeting

There have been no events of special importance during the past month. The most interesting was the meeting of the steel associations on October 15. There has been a lot of speculation as to what would happen at that meeting. For some time past the expectation had been growing that the prices of steel plates and sections would be reduced, and there was practically no forward buying taking place. It was known, too, that some of the steel makers were favourable to a reduction in price. However, the matter was discussed very thoroughly, and it was eventually decided to leave the controlled prices unchanged until the next joint meeting, which is to be held in January. After all, there is really no very good argument for a reduction. The prices are not excessive; indeed, many of the works find it impossible to make a profit at these prices, after the carriage, rebate and reduced output are taken into account. Moreover, a reduction would not have increased the orders to the steel works. It would have resulted in further financial embarrassment without any compensating benefit. A wrong impression has been given by the prices at which the controlled materials, such as billets, small bars and boiler plates are being sold. The continued depression has caused makers to sacrifice economic prudence by cutting prices without any regard to manufacturing cost. As a result we see bars under 3 in. round being sold at under £8 per ton, while for 3 in. round the association price is £9 7s. 6d.; and steel sheets  $\frac{1}{2}$  in. thick are offered at £9 5s. to £9 per ton, while the official price for  $\frac{1}{2}$  in. thick is £9 12s. 6d. As soon as trade revives these prices will advance, but at present the margin between controlled and uncontrolled is too glaring. Unless a revival in the steel trade sets in before the middle of January, and that does not look very probable, it will be difficult to resist the pressure both from inside and outside for a reduction in plates and sections. It is an open secret that one or two works would welcome absolute freedom

in prices, as they would then make a bold attempt to capture sufficient of the available trade to keep their mills at full output, and that would enable them to determine to what extent price cutting is justified by increased production. Such a step would be disastrous for many of the works, although it would have the effect of quickly weeding out the inefficient plants and leaving the trade to those who have kept themselves up to date all round.

#### Policy of French Works

The situation on the Continent has not yet cleared itself. The Steel Cartel has only just settled some of the differences between the constituent members, and the effect of this is seen here in the continuance of the extremely low quotations for all classes of steel. Steel billets are offered at £4 10s. to £4 15s., delivered into works in this country, and small steel bars are still about £5 per ton. The French works have been largely responsible for this. Their home trade has not suffered anything like that of the other European countries, but their export trade has declined considerably during the year, and they have therefore made a special attack on the only free market left to them. The lengths to which they have gone, aided by their comparatively good home trade, has alarmed the other members of the Cartel, who are insisting upon an increase in the French export prices. The margin between these prices and the British home prices is so great that whatever increase is likely to be agreed will not divert the trade from the Continent. The menace of foreign dumping is seen in the list of rebates which the German steel works give to the finishing and engineering industries on steel which is intended to be worked up and exported. These rebates represent the difference between the domestic market price and the world market price, and they range from 35s. per ton on blooms and billets to 49s. per ton on bars.

#### Low Steel Scrap Prices

The general slump in the steel trade has affected the price of raw materials, and in this respect has mitigated slightly the fall in steel prices. Steel scrap can be bought at under 50s. per ton, and is now within a few shillings of the lowest price reached within the last 50 years. The works, of which there are still a few, which work on cold charges, can take full advantage of this by increasing the percentage of scrap in the charge and so reduce the cost of the ingot. Blast furnace coke has also fallen to the pre-war level, but there it is likely to remain at present, as the coke makers will prefer to reduce their outputs rather than cut prices any further. Coal prices have remained fairly steady, and in view of the provisions of the new legislation it is not likely that there will be any reduction in the near future.

#### Pig Iron Market

The pig iron market has shown no life at all during the month of October. The fixed minimum prices in the Midlands have been maintained, but buying has been very restricted and outputs have had to be reduced. In the Derbyshire, Notts, Leicestershire and Northants districts, less than half the furnaces are working. In Cleveland also business is very quiet, but quotations remain officially the same. There is talk of a somewhat better tone in the hematite market, although there is still a tendency to hold off buying in the hope of still lower prices. In no section of the steel market is there any liveliness. Notwithstanding the low prices which are quoted, consumers are not inclined to buy forward. The mills are desperately short of specifications for plates and sections, and short-time working is on the increase.

The output returns for September show an increase over August, but this was to be expected, as the holidays in August, extended as they were on account of the state of trade, seriously diminished production. Nevertheless, the September figures show up badly when compared with last year. The output of pig iron was 425,000 tons, as against 664,600 tons in September last year. The output of steel ingots was 580,600 tons, compared with 847,900 tons in the same month last year.

**Some Inventions of the Month****By Our Patents Correspondent***Abstracts of other Patents of metallurgical interest will be found in our Patent Literature published weekly in THE CHEMICAL AGE.***Alloys**

A BRONZE ALLOY containing 7-13 per cent. of aluminium, 2-6 per cent. of iron, and the rest copper, is described in Specification 333,442, dated October 2, 1929, by G. Bärtges, of Duisburg, Germany. The metals are melted with slag-forming materials, e.g., alkalis or borax, and deoxidising agents, e.g., sodium, potassium, or magnesium, and the melt is poured. After solidification the mass is remelted under a covering of the slag formed by the first melt and with the addition of further deoxidising agents such as magnesium.

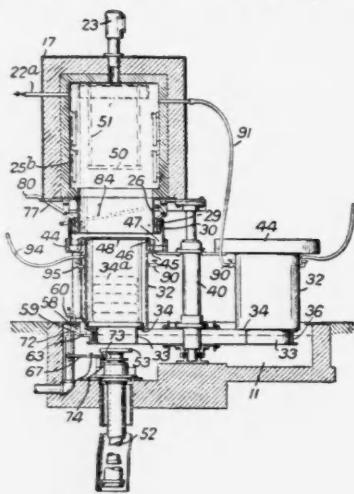
ALLOYS comprising 25-45 per cent. of copper, 5-15 per cent. of nickel, and the remainder iron, with or without an addition of chromium up to not more than 20 per cent. of the iron content, are described in Specification 333,774, dated May 30, 1929, by W. P. Digby, of London, the subject-matter being divided from that of Specification 322,216 (see THE CHEMICAL AGE, Vol. XXII, p. 13 [Metallurgical Section]). The metals may be melted in a gas, coke, or oil fired furnace, and the molten product finished in an induction furnace, or the whole operation may be performed in an induction furnace. Manganese may be added in small amounts five minutes before pouring, and a deoxidant such as magnesium immediately before pouring.

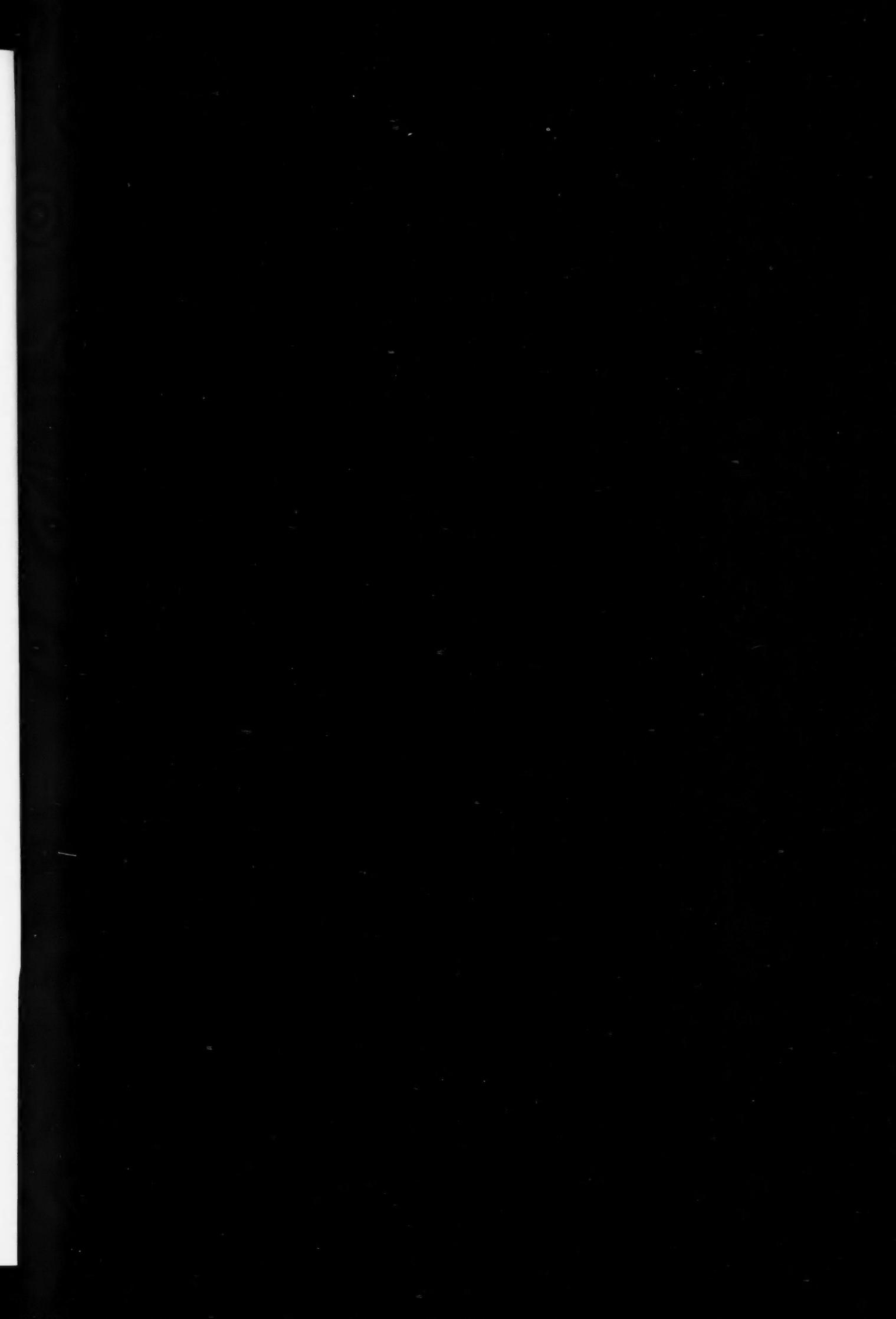
**Alloy Steels**

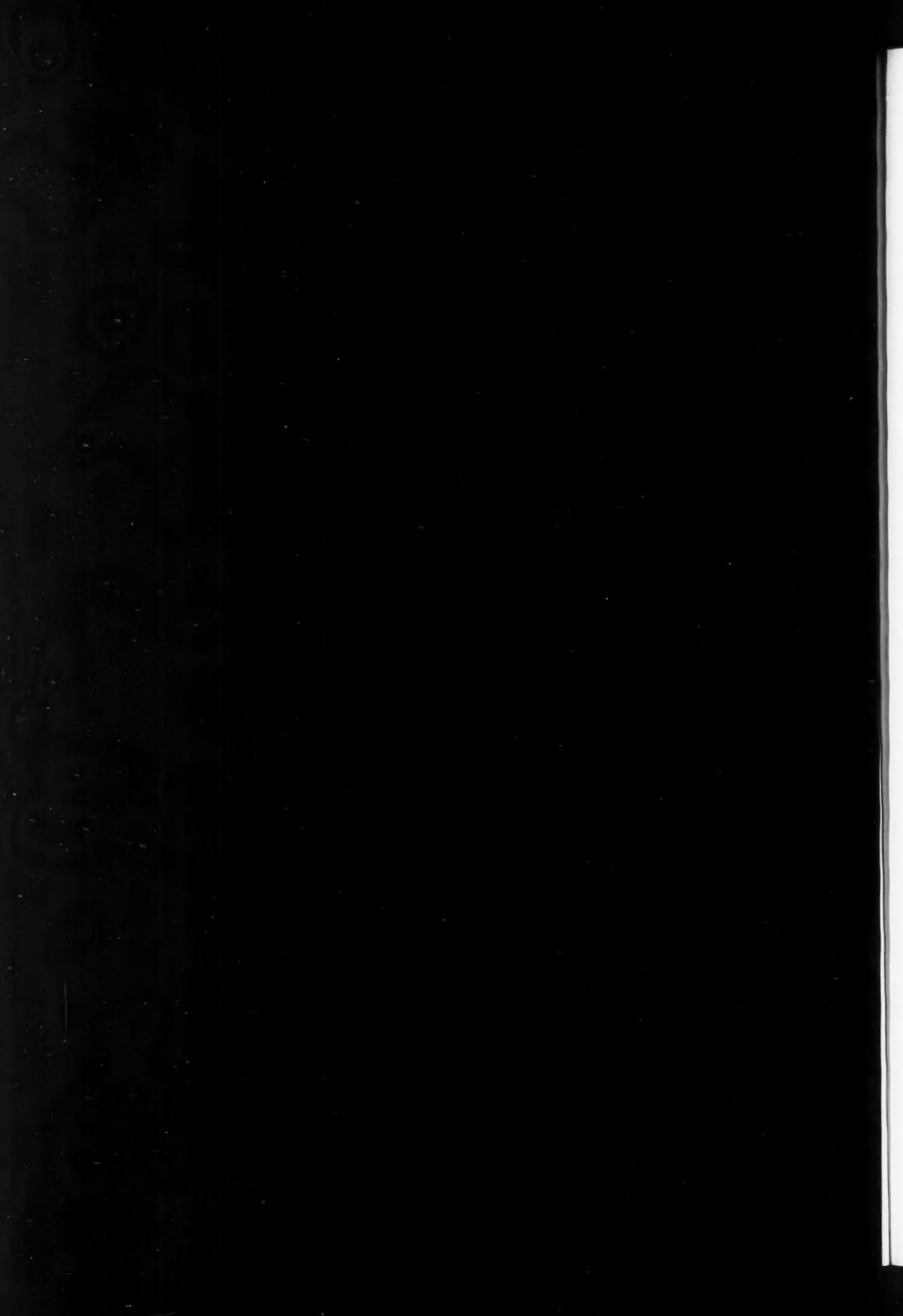
AUSTENITIC nickel-chromium steels containing 10-25 per cent. of chromium and 25-5 per cent. of nickel are polished or ground, heated to 100-400°C., and cooled quickly or slowly to render them incorrodible. The steels may also contain one or more of the metals molybdenum, tungsten, copper, aluminium, zirconium, vanadium, and titanium. See Specification 333,237, dated February 5, 1929, by W. H. Hatfield and H. Green of Sheffield.

**Annealing Metals**

APPARATUS for use in annealing metals in a non-oxidising atmosphere is described in Specification 332,436, bearing the International Convention date January 14, 1929, by C. F. Kenworthy, of Waterbury, Connecticut, U.S.A. The charge 34a is carried in a series of containers 32 adapted to be moved







# Monthly Metallurgical Section

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**NOTICE.**—Communications relating to editorial matter for our Monthly Metallurgical Section should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.

## Recovery of Metals from Waste Materials

By J. W. Hinchley, A.R.S.M., Wh.Sc., F.I.C. (Member)

Among the papers read at the Conference of the Institution of Chemical Engineers on "The Utilisation of Trade Wastes" on Thursday and Friday last, was one by Professor Hinchley on "The Recovery of Metals from Waste Materials." Besides the general account, reproduced below, the paper contained a detailed reference to the actual plant employed.

PROCESSES for the recovery of useful metals from waste materials have almost an ethical claim to consideration, on account of the fact that the available supplies in the world are not unlimited. The common metals are being recovered to a greater extent every year, and in spite of the present depression, through the unregulated industry of recent years, it is certain that waste must be still further reduced, or the value in that waste recovered, if we wish to maintain a high standard of civilisation without drudgery. When we learn to regulate our supplies, we shall realise the importance of conserving what we have, and wasting a minimum.

Waste materials containing metal may occur as (a) scrap machinery parts, turnings, cuttings and dross; (b) dumps of waste material from old industrial operations; (c) material recovered from town's refuse. Under the headings (a) and (b) the processes involved must give a definite commercial profit, but in the case of (c) the necessity of getting rid of the material may justify a loss if the health of the community and the amenities of the neighbourhood are to be improved.

Metal constitutes on the average 3·6 per cent. of the refuse collected in the London area by the various councils. Over 310,000,000 used tins, or 30,000 tons of tin plate, and, in addition, 13,000 tons of other metal are placed in refuse receptacles in London every year. The cost of disposal and collection is approximately £40,000. Only 40 per cent. of the tins are sold to steel smelters or to detinning firms, the remainder go to provide quarters for rat colonies in the huge dumps in Essex and Middlesex.

### Scrap Iron and Steel

Scrap iron and steel apparently present a simple problem, but with more careful sorting, and by the use of standardised materials, in the first instance, much greater values are capable of recovery than is the case to-day. The blast furnace and cupola are the ordinary means of dealing with the scrap; but the product obtained is very often of low quality. These furnaces are particularly convenient on account of the possibility of removing some of the impurities by volatilization or by suitable fluxing reagents. Some impurities, however, prevent the use of such scrap for the production of a high class metal.

The cost of sorting waste material is a very serious item in the cost of recovery, but this may be avoided entirely in large industrial undertakings by avoiding the mixture of materials in the first instance. Large quantities of metal can thus be handled for what it is, rather than as a raw material for the blast furnace.

With copper alloys the problem is more complicated, but on account of the greater value of the metals there is more incentive to careful separation or sorting. In the treatment of turnings and cuttings, water and grease are usually removed by heating the material in a continuous roaster having a travelling chain bed. A reducing atmosphere is maintained by means of oil burners, and the temperature obtained brings about the evaporation of the moisture, and the combustion of the oil that is present. After the material has cooled the removal of any adventitious iron by a magnetic separator is readily made.

The briquetting of turnings and cuttings is a subject of some controversy, and several firms have ceased to briquet such material. There are advantages in briquetting, such as

ease of handling, and, of adding fluxes, but the most careful tests fail to support the claim that the efficiency of recovery is improved. The easy addition of fluxes such as potassium sulphate, borax, sodium carbonate, ammonium chloride, etc., for the removal of aluminium, sulphur, etc., is a valuable advantage. The development of standardisation has reduced the number of bronze alloys considerably and enabled the successful sorter to obtain higher values. The efficient melting of cuttings and turnings involves care in obtaining the development, in the first instance, of a good bath of molten alloy into which the briquette or loose material may be immersed, thereby preventing serious oxidation.

### Successful Electrolytic Process

In spite of standardisation and careful sorting, melting methods fail to give satisfactory materials for the manufacture of a good bronze, owing to the presence of extraneous metals such as lead, antimony, zinc, etc., while metallurgical methods fail to produce pure copper or tin from them. O. Scarpa \* has developed an electrolytic process which has been in successful use for several years on the Continent. The scrap bronze is cast into anode plates 3 cm. thick and over a square metre in area. Thin sheets of copper form the cathodes and a hot solution of copper sulphate containing free sulphuric acid is circulated through the bath. The E.M.F. is 0·4 volt and a thickness of 2 cm. of copper is deposited on the cathodes with a current efficiency of over 90 per cent.; the energy consumption being 0·665 kilo-watt-hrs. per kilo of copper deposited. The purity of the copper is 99·97 per cent., and the slimes removed from the bath usually contain about 25 per cent. of tin, 8 per cent. of copper, 7 per cent. of lead, 3 per cent. of zinc, 0·3 of iron, 0·5 per cent. of antimony, with traces of other substances.

Almost all the tin is found as stannic and meta-stannic acids in the slimes of the electrolytic bath, but a small amount of colloidal tin oxide is produced, and forms one of the difficulties of the process. A small amount has a favourable effect on the deposition of copper by improving the grain, but a larger amount brings about black impure deposits. By hydrolysis of the sulphate of tin, which is first produced, the colloidal oxide and free sulphuric acid are formed, so that the solution must be continually purified. In practice, a portion of the electrolyte is shunted through a re-conditioning plant, consisting of a coagulating bath in which the liquor is heated to flocculate the tin oxide, a decanting device to remove this oxide and a column containing copper cuttings, and through which air is blown, for the neutralisation of the free sulphuric acid and the enrichment of the liquor in copper.

About 95 per cent. of the tin is extracted from the slimes by treating them twice with hot caustic soda solutions, using the stronger solution last. The solution of stannate of soda, obtained after purification for the removal of zinc, lead, etc., is also treated electrolytically, and the tin deposited on thin tin cathodes, the anodes being of iron. The spongy tin obtained is very pure, and, after washing, is melted in reverberatory furnaces. The consumption of energy is 10 kilo-watt-hours per kilo of tin obtained. The residue of slimes is reduced in a reverberatory furnace and the alloy obtained retreated electrolytically. Problems connected with the

\* (Brit. Pat. 1925, 227,125)

silver and gold found in the final residue and the by-products, the sulphides of zinc and lead, still remain.

**Tin and Iron Recovery from Tin Plate**

The universal use of tin plate containers presents important metal recovery problems; the utilisation of the scrap obtained by the container makers and the disposal of the used container. Formerly tin plate often contained 5 per cent. by weight of tin, but just before the war this figure had dropped to 2 per cent., and now, owing to the improved methods, is usually little more than 1 per cent., whilst the protective value of the coating has not been diminished.

Since the steel smelter will not handle material containing more than a trace of tin, the treatment of factory scrap is not easy, while the treatment of old tin cans contaminated with paints, oils, varnishes, foods, drugs, etc., presents still further difficulties. Hundreds of patents have been taken out for processes during the past 70 years, and modern processes are all improvements on abandoned processes of the past. Successful processes of to-day may be classified as:—(a) Electrolytic; (b) dry chemical; (c) wet chemical.

Although acid electrolytic processes are known, the usual electrolyte is a 70 per cent. solution of caustic soda heated to 70° C. Iron plates, which are removed every 12 hours or so, form the cathodes, while the scrap tin cuttings, supported in iron cages about 15 in. wide, form the anodes. The tin is deposited as a spongy grey mass, and after being scraped off the iron plates, washed and pressed into small briquettes, is melted and cast into ingots. The de-tinned contents of each iron cage are charged into a hydraulic press and formed into a briquette of convenient size. Any oil, grease, paint or varnish on the scrap is attacked by the caustic soda, forming a soap which dissolves in the bath, and to some extent preserves the spongy tin from oxidation on its removal from the bath. Complete stripping is not always obtained by this process, owing to faulty penetration of the solution to the centre of the cage. On account of the exposure of the bath to the air, the caustic soda must be regenerated at frequent intervals by means of lime. A great advantage is that the tin is recovered in the metallic state. The energy consumption is about 100 kilo-watt-hrs. per ton of scrap treated.

**Dry Chemical Processes**

In dry chemical processes chlorine is the agent usually used, although dry hydrochloric acid, fused caustic soda, and other salts have been proposed. The first patent for a chlorine process was taken out in 1850, but it was not until 1905, when Goldschmidt overcame the difficulties of large scale work and cheap chlorine was available, that the process became successful. To-day three-fourths of the tin scrap recovered throughout the world is treated by this process. The scrap is pressed into bundles, packed into towers, and dry chlorine mixed with air admitted at a pressure of two atmospheres. The process originally took 30 hours, but the time of treatment was reduced to 15 hours by using as a solvent excess of stannic chloride, which drained off from the bottom of the tower. Other solvents, such as carbon tetrachloride, have been proposed.

The great disadvantages of the process are the use of a poisonous gas, the necessity for the exclusion of both water and organic matter, the drying of both the gas and the charge and the previous cleaning of the charge by the removal of any varnish, grease, paint, etc. The heat of formation of stannic chloride is high, and if the temperature be allowed to rise the iron may be attacked and the whole mass raised to incandescence. On the other hand, large quantities can be treated at one time, chlorine is cheap, the residual scrap is well stripped, and the stannic chloride is pure and in great demand. A plant to treat 200 tons of scrap per week would cost about £16,000; 80 to 100 lb. of chlorine being used per ton of scrap treated, the cost of treatment is about equal to the value of the de-tinned scrap. Moist chlorine has also been proposed; hydrated stannic chloride being used as a solvent.

**Wet Chemical Processes**

Of wet chemical processes the only successful ones have depended in some way on caustic soda as the solvent. Although caustic soda solution will attack tin, the boiling solution is too slow; more than two days being required, and then the scrap is not well stripped. The reaction can be speeded up by means of air, but oxidising agents in the solution gave the desired result with certainty. The use of alkaline solutions of chromates, nitrites, sodium chloride, sodium nitrate, manganese dioxide and litharge have been patented.

The litharge alkaline process patented in 1884 was successfully at work in Germany in 1890. It fell into disrepute as the electrolytic alkaline process increased in popularity. It was again patented by Ramage in the United States in 1911, the tin being separated from the stannate of soda solution by scrap galvanised iron. The alkaline nitrate process has been developed in the United States by the Goldschmidt Co., but it is difficult to see what advantages this process possesses. A recent improvement in the lead alkaline process has been worked out by the author and a simple continuous cyclic process obtained which offers advantages over other processes and has few drawbacks.

The early workers added such an amount of massicot or litharge to a hot strong caustic soda solution that some of the litharge remained in suspension. This solution, near its boiling point, dissolves the tin from the tin plate, and deposits lead in a finely divided condition. A solution of sodium stannate is obtained from which the tin may be precipitated, in the metallic state by zinc, as metastannic acid by carbon dioxide, or as calcium stannate by means of lime. The finely divided lead may be oxidised by the usual methods and again dissolved to form a treating liquor.

Unfortunately, the chemistry of lead in an alkaline solution is very complicated and it is easy to obtain an insoluble lead oxide with consequent commercial loss. The nature of the solution of lead hydroxide in caustic soda is a matter of controversy, and careless manipulation, on a commercial scale, may lead to the precipitation of lead oxide in an insoluble form. The solution in dilute alkali is very different from that in concentrated solution. According to the strength of solution and the temperature, lead may be deposited as lead oxide in a very insoluble form.

**Modern Treating Liquor**

In the latest process, more suitable solutions are obtained, and regeneration of the original liquor is readily accomplished. The treating liquor may be obtained in the first instance by dissolving lead hydroxide, precipitated from lead acetate by molecular proportions of sodium hydrate, in caustic soda solution of the requisite strength or by treating spongy lead obtained in the process of detinning with caustic soda solution in air.

This treating liquid consists of 4·42 per cent. lead, 10·25 per cent. caustic soda and 85·3 per cent. water, all by weight or in gram molecules 1 Pb, 12 NaOH : 185 water. This solution, either hot or cold, is very stable although very active. At a temperature of 90° C. the tin is removed, by this solution, from the tin plate in a few seconds and until the lead in the solution is nearly all displaced the rate of attack is high. In this reaction, for every two atoms of tin dissolved three atoms of lead are deposited, so that the following equation does not completely indicate what occurs.



A solution of stannous hydroxide in excess alkali is a very powerful reducing agent, and there is little doubt that it becomes oxidized to stannate with a further deposition of metallic lead. The reaction is also interesting in that below a temperature of 56·7° C. the rate of reaction is negligible. At temperatures above 56·7° C. the rate increases as the temperature increases. A temperature of 90° C. for the treating solution is used because the rate of evaporation is of definite value and the rate of reaction is as great as is desirable.

On account of the deposition of finely divided lead it is necessary to agitate the tin plate to ensure its removal from the surface of the material, and revolving drums are used so that the process becomes a continuous one. The sodium stannate solution obtained is treated with caustic lime, by which a precipitate of calcium stannate is obtained and the caustic soda solution regenerated for use. The precipitated lead is placed in a tower with the regenerated caustic soda and air blown through. After 20 hours the original treating solution is obtained by the hydration of the lead and its solution in the liquor.

The author concluded with a reference to the treatment of dumps of ashes, clinker, slag, broken crucibles, etc., which are often found in the vicinity of old works, and may sometimes be profitably treated for the recovery of their metal contents. He expressed his thanks to Messrs. S. G. M. Ure, A. H. Lovelless and J. D. Parsons, who had all contributed to the work included in the paper.

## Destruction of Chrome Nickel Heating Elements

### An Investigation into the Causes

The following is abstracted from a contribution by the Research Department of the Steatit-Magnesia A.G., Berlin, entitled, "The Causes of the Destruction of Chrome Nickel Heating Elements." It is shown that, contrary to general belief, the wires themselves, and not the ceramic supporting materials, are the cause of the trouble.

THE causes which bring about the destruction of the nickel chrome and other resistance wires used for electrical heating have been studied by reproducing the most unfavourable working conditions, and taking observations of the result of deliberately overloading the resistance wire until it burnt out. Both chemical and electrical phenomena have been found to contribute to the production of the well-known coloured deposits and to the local overheating which causes the melting of the wire. Some surprising facts have been established, such as the formation of chromium trioxide by purely thermal reactions and the migration of the oxide mist so formed in the electrical field.

The majority of electrical heating apparatus have as the source of heat resistance wires or strips consisting of an alloy of chromium and nickel, or chromium, nickel and iron. The heating element is in some cases carried on mica supports, but more usually on formers of pressed ceramic materials. The electrical heating industry, however, is constantly confronted by the fact that such heating elements burn out under the most unexpected conditions and in a quite incalculable way. As a rule, the ceramic materials are held to be responsible for this. The opinion has been expressed that the ceramic material attacks the heating wire under the influence of the electrical current, or is itself ruptured electrically owing to the diminution in its dielectric strength on heating. The occurrence of coloured deposits on the ceramic carriers is attributed to electrolytic action between the ceramic material and the wire. Arising from this belief, great importance has been attached to the insulating qualities of the material at high temperature, and the opinion formed that the smaller the shunt currents flowing through the carrier itself when heated, the more reliable is the material for the purpose.

#### Reproducing Conditions of Practice

Actually, however, it has been shown that the question is by no means so simple. It should be remembered that compounds of silicates and refractory oxides, substances which at high temperature conduct partly electrolytically and partly electronically, are in contact with glowing metals, which on their part at high temperature form and throw off conductive oxide crusts. On the other hand, it is found that the "burning-through" phenomena on ceramic carriers of quite different composition are practically the same. The measuring of the insulating resistances of different materials also shows that the ceramic substances which have been found most satisfactory in the electrical heating industry are not always those with the highest insulating qualities.

During an investigation into these destructive reactions it was found possible to produce in the laboratory the phenomena encountered in practice, and study these in detail.

If a heating element, e.g., a bowl fire former, which has been in use for some time, is dismantled, it is often found that there are coloured deposits on the surface of the ceramic former following the outline of the heating wire. The colour of these deposits is brown or brownish-green. Occasionally, one finds in the vicinity of these silhouettes diffused deposits of a lemon-yellow colour. The first part of the investigations was devoted to the question as to whether there is a connection between the conductivity of the ceramic material and these coloured deposits. The results of the chemical analysis were as follows:—

The yellow deposits contain chromium trioxide ( $\text{CrO}_3$ ) in the anhydrous or hydrated condition. The presence of chromic acid ions is at once detected by the formation with silver nitrate of the red silver chromate  $\text{Ag}_2\text{CrO}_4$ . The potassium iodide starch reaction (precipitation of free iodine) and the reaction with sulphurous acid (formation of green  $\text{Cr}_2\text{O}_3$ ) were also successful. If these deposits are heated above  $250^\circ$ , the yellow colour disappears, due to dehydration and decomposition of the chromium trioxide. Nickel and iron are also present in the yellow deposits in the form of oxides, sometimes in only very small proportions. The

presence of nickel can be proved with Dimethylglyoxime, and iron by the well-known red colouring with Ammonrhodanid. The brown and brownish-green deposits consist of oxide of iron, nickel and chromium. It can be assumed that the nickel oxide formed at the same time as the chromium trioxide combines with this to form nickel chromate. This substance is stable up to nearly  $500^\circ$ . As the deposits examined were completely dispersed or decomposed above  $250^\circ$ , it can be assumed that the chromic acid is either not at all, or to only a slight extent, combined with nickel. The iron reaction can be obtained only after previous treatment with concentrated acids, an indication that the iron is present in the deposits as  $\text{Fe}_2\text{O}_3$ , insoluble in water, and is also not combined with chromic acid.

#### Yellow and Brown Deposits

It was then necessary to ascertain whether these coloured deposits were caused by reaction between ceramic material and heating wire, i.e., whether the ceramic substance in any way causes or assists oxidation of the metals in the wire. If one places some chrome nickel wire on a white ceramic support and makes the wire glow strongly, the brown deposit is left at the places in direct contact with the wire, and this is surrounded by the yellow deposits. It is found sometimes that these yellow deposits extend past the end of the glowing resistance wire, that is to say, where electrolytic actions between the materials cannot have taken place. Whether the ceramic material plays any part in the formation of these deposits is very simply ascertained by the following experiment.

A glowing chrome nickel wire was stretched in the air between two points in front of and at 1 mm. distance from a ceramic plate under conditions in which direct contact was impossible. After a few minutes a yellow diffused "silhouette" of the wire was formed on the plate. The temperature conditions for the formation of deposits was also investigated. The lowest temperature at which definite evidence of the presence of chromic acid could be obtained was  $900^\circ$ . After heating the wire for one hour at this temperature, with the plate at 1 mm. distance, clear evidence of the presence of chromic oxide was established by means of a silver nitrate test. On increasing the temperature, the rate of deposit increased very rapidly. At temperatures from  $1,100-1,150^\circ$ , wide yellow strips of deposit were obtained in 2 mins. In the centre of these strips, however, there was generally found a line of the described brown colouring. The tests were then repeated with a silver plate to catch the deposit instead of one of ceramic material. The results were the same, and the presence of chromium trioxide was definitely established in this case also. This proves that the ceramic material has nothing to do with the formation of the coloured deposits, but acts merely as a collector for the oxidation products of the wire.

#### Production of Ozone

Above  $900^\circ$  C. the chrome nickel wire produces large quantities of ozone from air. The characteristic smell of ozone can be readily detected in the vicinity of the glowing wire and its presence confirmed by the usual chemical tests. If one places the glowing wire in a glass tube and passes a slow stream of air over the wire and over a filter paper with Arnhold's reagent (an alcoholic solution of tetra-methyl-diaminodiphenyl-methane), the paper shows the usual violet colour. There is an obvious connection between the presence of this ozone and the formation of the chromium trioxide.

The whole process is probably this: The glowing wire throws off a certain amount of vapour or of ions of the metal and the atoms or ions thrown off are oxidised in the ozonised air layer around the wire. A very fine separated oxide mist forms in the cooler neighbourhood of the wire and settles on the nearest solid article. To bring about the formation of the yellow deposit there must be relatively cool collecting surfaces near the wire. If the temperature of the collecting plate has risen above  $250^\circ$ , the chromium oxide decomposes

into chromium sesquioxide, and the dark deposits are formed. We were unable to decide with our experimental apparatus whether all chromium sesquioxide found on the ceramic plate was formed from chromium trioxide or whether it was formed direct. The yellow deposits are formed more readily on porous than on dense plates. Apparently the porous plate on account of its smaller thermal conductivity offers cooler collecting surfaces, especially on the edges of the material.

The examination of burnt out heating elements with spiral wire coils in some cases brought to light pieces of wire in which two neighbouring windings were firmly melted together. It was to be clearly seen that the metal had temporarily fused at the end of the winding. At certain points, therefore, the wire had attained a temperature of at least  $1,250^{\circ}$ . It is not impossible that the temperatures reached were much higher, for the melting points of wires of chrome nickel alloys go up to over  $1,400^{\circ}$ . Investigations were then made to ascertain in what way the high temperature was attained at these points. If short circuits are deliberately produced between adjacent turns of the wires of a heating element it will be clearly seen that the wire glows more brightly at these points than elsewhere. The shunt current from winding to winding can only be of small magnitude on account of the high contact resistance and the small voltage difference, but this is concentrated into such a small conductor cross-section, that the temperature can exceed that of the glowing wire by several hundred degrees.

#### *Conclusions from Tests*

At these points a clearly perceptible and progressive fusion of the wire sets in until it eventually burns out. A small arc forms and the wire is melted and simultaneously oxidised. There are now two things that can happen; the melted metal can make contact with the neighbouring winding and thereby avoid breakdown; the point recools and the resistance is again ready for use. Otherwise the fusion continues. Oxides of chromium, nickel and iron are formed. These are conductive at high temperature and prevent a quick extinguishing of the arc. The heat now becomes so great that the oxides react with the carrier and form a dark-coloured clinker (or slack). After the arc is finally extinguished, black spots are left which by fusion have penetrated the material deeply and in the case of thin-walled tubes, sometimes extend right through the wall of the tube.

The general result of the tests described may be stated as follows: The well-known burning out of heating elements, a frequent source of trouble, is caused by the wire. Heating wires must obviously be treated much more carefully and considerately than is generally the case. The coloured deposits occurring so extensively without the chemical or electrolytic co-operation of the ceramic material serve as a proof that overheating has taken place. Where it is a question of the supply of radiating heat, a glowing temperature of  $900^{\circ}$  should be sufficient, and resistance wires and strips heated to much lower temperatures should be employed where only low temperature heating is required. If an arc occurs on the burning out of the heating wire, the ceramic element is also injured by the action of the oxides. The actual conductivity at high temperature of the ceramic material has much less importance than has hitherto been assumed.

#### *Canadian Nickel Refining*

THE International Nickel Company has awarded to the Dominion Bridge Company of Montreal a contract for steel work amounting to about £100,000 in connection with the new plant at Sudbury, at which nickel and copper matte will be separated. The total cost of this further addition to the company's programme will be about £400,000 and the new plant is to carry out that portion of the refining process by which nickel is separated from the copper content in the Bessemer matte produced at the Copper Cliff smelter.

At the present time matte is despatched from Copper Cliff and Coniston to the company's plant at Port Colborne, Ontario, where nickel is refined, the blister copper being sent back to Copper Cliff for treatment in the new plant of the Ontario Refining Company, Ltd. The new structure will use the equipment of the No. 1 refinery unit, which is now being transferred to Copper Cliff from Port Colborne. A year's work will be taken up in the re-installation of this equipment, 200 men being employed on construction work.

#### *The Estimation of Cobalt in Ferro-Cobalt*

*(From a Correspondent)*

THE best method for the estimation of cobalt in ferro-cobalt seems to be the electrolytic method. The procedure is similar to that employed for nickel and may conveniently be carried out as follows.

Dissolve half a grain of drillings in the smallest possible quantity of nitric acid, and when solution is complete add about 20 ccs. of 50 per cent. sulphuric acid and evaporate to fumes of sulphur trioxide. In order completely to eliminate nitric acid, which would interfere with the electrolysis, the solution should be allowed to fume strongly for about ten minutes. Cool and dilute with 20 ccs. water. Heat the solution to nearly boiling and pass hydrogen sulphide to precipitate copper and arsenic. Filter and boil the solution in order to expel hydrogen sulphide completely. Add 2 ccs. hydrogen peroxide to oxidise ferrous compounds to ferric state and then add ammonium hydrate until slightly alkaline and boil. Filter off the ferric hydroxide and wash with water containing a few drops of ammonia. Redissolve this iron precipitate in a small quantity of dilute sulphuric acid, adding a few drops of hydrogen peroxide to keep the iron oxidised, and precipitate as before with ammonium hydrate. Combine the two filtrates.

In presence of comparatively large amounts of iron the basic acetate separation of iron is necessary instead of the double precipitation with ammonia as above, as ferric hydroxide occludes some cobalt.

The solution containing the combined filtrates is made ammoniacal with 50 ccs. of strong ammonium hydrate and electrolysed as usual. If possible some method of agitating the solution during electrolysis should be employed. The solution should be tested for complete deposition by mixing a drop with a few drops of colourless ammonium sulphide. When no dark coloration is produced the electrolysis is finished. The cathode is washed in water, then alcohol, dried and weighed, and the increase in weight gives the cobalt on  $\frac{1}{2}$  gram of sample.

It should be noted that any nickel which may be present will be deposited with the cobalt, and should be estimated separately by the dimethylglyoxime method and deducted from the weight of the deposit.

In cases where an electrolytic apparatus is not available, the best alternative is probably the phosphate method. To do this, weigh down one gram of sample and proceed as above to the stage when the filtrate is obtained from the double iron precipitation. Now make faintly acid with acetic acid and then make up to 500 ccs. and take 100 ccs. equal to  $\frac{1}{2}$  grams sample. Heat the solution to boiling and add a solution of ammonium phosphate containing about ten times the amount of cobalt likely to be present, stirring vigorously during the addition. The bulky violet precipitate which is at first formed gradually changes to a pink dense crystalline precipitate. This is allowed to settle, filtered off, and after washing well is ignited carefully to cobalt pyrophosphate which contains 40.41 per cent. cobalt.

#### *Errors in Molybdenum Analysis*

SEVERAL cases in which widely divergent amounts of molybdenum were reported by different responsible customs laboratories, to which quartered portions of the same pulp samples had been sent for analysis, have recently been investigated by the Rare and Precious Metals Experiment Station of the United States Bureau of Mines, in co-operation with the University of Nevada, Reno, Nevada. An oxidized lead ore which contained considerable vanadium and less than a per cent. of molybdenum was reported as containing molybdenum trioxide ranging from 3.14 to 0.24 per cent. The high results were due to the non-complete elimination of vanadium from the molybdenum.

A fine grained rock containing considerable graphite did not indicate clean residues by the usual acid decomposition. Several chemists used prolonged fusion with soda ash and nitre. The solutions contained phosphorus derived from the ore which was precipitated as lead phosphate and mistaken for the molybdate. The elements most likely to cause trouble are vanadium, tungsten, uranium, arsenic, antimony, titanium, tin, phosphorus and chromium.

## Metallurgical Topics: Monthly Notes and Comments From Our Own Correspondents

### Non-Ferrous Metals in Chemical Engineering

DR. RICHARD SELIGMAN, the president of the Institute of Metals, gave a lecture on November 13, at the Royal School of Mines, and before the London Section of the Institute, on the use of non-ferrous metals in chemical engineering. He spoke on the design of plant, and the temperature conditions which metals have to encounter in chemical factories, and the heavy loads they have often to withstand, and he also dealt with corrosion problems, not only from the point of view of the lasting properties of the metals exposed to corrosive influences, but from that of the possible contamination of the product from the formation of noxious adulterants. He then dealt specifically with the use, in chemical plant, of lead, tin, copper, platinum and silver. He stressed the admirable properties of nickel, but pointed out that, owing to its absorption of gases at elevated temperatures it often became almost unworkable. Aluminium had very many valuable applications but its proneness to exaggerated grain growth and the formation of large crystals required to be guarded against when annealing followed on the cold working of the metal.

### The Nitriding of Steel

IN view of the steadily increasing interest which is being taken in nitrogenising, nitriding, nitrarding or nitrating steel (the nomenclature of the process is still somewhat indeterminate) it is interesting to review the literature of the influence of nitrogen on iron, and on nitrogen-iron alloys and to note how greatly opinions have altered within the last 25 years. The view that the effect of nitrogen was purely baneful been relegated to the past. The work of Fry helped to elucidate the earlier work of Braune, Andrew, and others, while Bramley and Turner, in Part IV of their very detailed investigations on the gaseous cementation of iron and steel, published in the Carnegie Scholarship Memoirs of the Iron and Steel Institute in 1928, investigated the influence of various mixtures of carbon monoxide and ammonia, at different temperatures, on "Armco" iron—the usual basis for research work on iron—and found that the "carburising" properties of mixtures containing ammonia were much stronger than those of carbon monoxide, either alone or with admixture of hydrogen. When nitrogen was substituted for ammonia less nitride is formed during cementation, and it was also found that the presence of nitrogen completely altered the microstructure of iron-carbon alloys, and that, at above 800° C. marked segregation of the cementite occurred. Many other investigators have worked on the subject.

### Rationale and Applications of the Process

THE latest researches available were undertaken by Mr. B. Jones, of the Department of Metallurgy, University College, Cardiff, and form the subject of a doctorate thesis which has, moreover, been awarded a special prize in an open competition inaugurated by a contemporary (*The Iron and Steel Industry*, December, 1930). It is a remarkable piece of work, one of the kind in which this country has not hitherto, unfortunately, been as prolific as France, Germany or America, an example, that is, of a detailed scientific investigation directed to promoting technical progress. Mr. Jones finds, in effect, that not all classes of iron and steel can be beneficially subjected to nitrarding. In those in which it may be advantageously employed the after treatment is very important, as untempered specimens display great brittleness and loss of tensile strength. This, of course, bears out the older views as to the noxious effects of nitrogen, in steel. What is new is the discovery of means of obviating these and deriving important advantages from proper control. Mr. Jones also finds that the presence of nickel and of chromium favours the best results obtainable by nitrogenisation. He describes, too, what may be called a duplex process, calorising and nitrogenising as well. Such a process is, however, of no benefit to austenitic steels, nor to high chromium, nickel-chromium, chromium-vanadium or chromium aluminium steels, but is of distinct benefit to plain carbon, nickel, nickel vanadium, and nickel-chromium-vanadium steels when a large increase of hardness is induced by the double process. By calorising, it is found that certain

steels which would not otherwise yield to nitrogenisation could be nitrarded, with the resulting valuable case-hardening effects.

### Metallurgy and Steam Engineering

THE conceptions of the engineer are always a little ahead of his ability to give them formal expression; the principles he applies are applicable only within the limitations of the materials with which the metallurgist is able to supply him. The aeronautical motor developing a unit of horse-power per pound weight cannot materialise until the metallurgist places in the hands of the engineer a metal or an alloy capable of enduring temperatures and stresses beyond those which the metals and alloys at present at his disposal can safely withstand. The demand, to-day, is for materials exhibiting, in the highest degree, tensile strength, workability (by which a whole host of operations are nowadays to be understood) corrosion resistance and heat resistance. Duplex metals, by which is meant the core of one with an exterior of some other may serve in some cases provided the external protecting metal will stand up to the requirements, but in the main, a homogeneous metal is necessary, while, in regard to stationary steam or gas driven engines, where the element of massiveness detracts only on the score of prime cost, from the maximum efficiency, in the industrial sense the homogeneity must be both physical and chemical. Hence the greatest interest attaches to all research work on high heat high tensile metals and alloys the creep strength of which may prove equal to long-continued stresses of a diverse kind, undergone at relatively high temperatures.

### Conditions which have to be Met in Practice

THE critical pressure of steam is 3,160 lb. per sq. in., but this accompanies a comparatively low critical temperature. There are plenty of materials that, even at that pressure and temperature, could be relied upon, and, indeed, the Benson boiler is designed to work in that condition. It has not, however, made much progress, and it is evident that the direction along which high-pressure high-temperature steam practice will develop is in the utilisation of higher temperatures still, but with pressures not exceeding 2,100 lb. per square inch. The nearest practical approach to this is that attained in the Loeffler boiler. Three plants, one in Vienna, one in Berlin, and one in Vitkovice, Czechoslovakia, are now working on this principle, employing temperatures of 930° F. (500° C.), and pressures of 1,850 lb. per sq. in. This has only become possible by using special steels, and for the parts most subject to these intensive conditions it is understood that molybdenum steels are being used. Attention is, as a matter of fact, being intensely focussed on high endurance high corrosion and heat resisting materials, both at home and abroad, and a good many kinds of steel are being experimented upon. At these high temperatures corrosive influences that can ordinarily be ignored, make themselves felt and even pure water exercises corrosive action, particularly at the pressures developed. On the subject of heat and corrosion-resisting materials a very valuable memoir has just been published by the Iron and Steel Institute, from the pen of Dr. Valenta, of the famous Skoda works. It deals exclusively, however, with high chromium, high carbon cast iron, and with these alloys with aluminium as an additional element, and further, only with the constitution of these alloys and with their scale-resisting, rather than with their creep-resisting and tensile characters.

### Cosmetics and Metallurgy

IT has more than once been alleged—and not only by distinguished speakers at dinners of Metallurgical Institutions—that it was to the use of cosmetics of mineral origin that, in prehistoric times, the discovery of the art of smelting metals was due. The legend runs that some irate husband in the distant past made a bonfire of his wife's "beauty box" and thereby reduced its metallic contents "in contact with carbon" with the result that metal products were subsequently discovered amongst the ashes of the connubial

hearth. The use of mineral powders as toilet adjuncts is, of course, extremely ancient, and colour is lent to the hoary legend by some recent discoveries made, by Professor Raymond Dart, of the Witwatersrand University, in Northern Rhodesia, and described recently in *The Times*. Near Broken Hill there was found the remains of an ancient manganese mine, the first of its kind to be discovered, and it can hardly be imagined that the deposits were worked for their metallurgical value. Dr. Raymond tells us, as other scientists have told us before, that manganese, in the form of the black crystalline pyrolusite, was used for dusting the hair, in ancient times, just as red ochre was used for imparting the fashionable tint to the skin, and powdered green malachite was used, mixed with unguents, for painting the eyelids. To this list may be added antimony compounds, such as the sulphide, used as a depilatory, while many other instances will occur to those familiar with the composition of the cosmetics of the modern beauty shop. It may well be that to the accidental or purposive burning of some of these that the origin of the art of metallurgy in ancient times is to be ascribed, the forest fires of Lucretius notwithstanding. What has yet to be proved is that it is to the use of cosmetics by women alone that the metallurgical arts are to be ascribed, for ancient man was not without his vanities, and his prehistoric "vanity bag."

### International Aluminium Competition

AN annual competition, open to inventors of any nationality, has just been inaugurated by the International Aluminium Bureau to encourage the development of the aluminium industry. It will be divided into two sections, each with a prize of 25,000 French francs, the first devoted to proposals relating to the manufacture of an article or machine in aluminium or aluminium alloy, and the second dealing with improvements in the methods of working or using aluminium or its alloys, such as soldering, insulating, plating, etc. In addition there is to be a special prize of 50,000 French francs for the author of a suggestion which has exceptional interest or value under either section. The entries must contain a detailed description of the article or process and should give sufficient data to admit of experimental trials to enable the claims to be verified. The opening date of the competition will be January 1, 1931, and entries must be forwarded, in duplicate, to the Bureau International de l'Aluminium, 23, bis Rue de Balzac, Paris, 8e, between that date and April 1. In order to protect their ideas, competitors may apply for Letters Patent covering their invention, provided that their application occurs after the opening date of the competition. The bureau, on its part, undertakes not to divulge publicly the contents of the manuscripts submitted for the competition, which have not received an award, so long as they remain in their possession. The act of submitting a proposal to the Bureau for this competition is to be regarded as constituting an offer of the invention for sale, in the first instance to the bureau and then to any of the Associated companies, on terms and conditions to be mutually agreed. In the event of the Bureau or its associated companies refusing the offer, or their inability to agree on a price for the acquisition of the invention, the author can resume his rights, whether or not a patent has been taken out.

### A Really Rare Metal

THERE are a number of metals known to the scientific world as "rare metals," but few of them have been produced in so small a quantity as columbium, according to the United States Bureau of Mines, Department of Commerce. Except for about one-half ounce of metallic columbium made in 1906, the metallic columbium exhibited recently by a Chicago firm at the twelfth exposition of chemical industries represents the world's supply, consisting of 25 pounds of the metal, in sheets, bars, rods, and wire.

For all of its scarcity, columbium is not particularly valuable, as it has as yet found little or no industrial application. The properties of columbium resemble very closely those of tantalum. It is inert to nearly all chemical action and is soluble in a mixture of nitric and hydrofluoric acids. It readily absorbs gases by occlusion and has been patented as a "getter" in vacuum tubes. It may be rolled, drawn, hammered, formed, or cut with ordinary working tools. It welds readily to itself and other metals by the spot-welding process.

Columbium has received comparatively little attention, perhaps because of the difficulties in the methods available for its extraction. The adoption of a satisfactory method of separation would not only bring into use one more of the rarer metals but would bring about a cheaper production of tantalum.

### The Cornish Tin Mines

SPEAKING at the annual dinner of the Royal Geological Society of Cornwall at Penzance on November 22, Captain F. C. Cann, manager of Geevor Mine, St. Just, which is one of a number of others closed by the tin slump, said Cornish mining men had no need to be pessimistic, as he believed that next year they would see a big improvement and their mines would be flourishing again. Although he did not expect they would get back to the prices they had two or three years ago, they would nevertheless see prices which would make mining remunerative, and those mines which were worth restarting would be restarted. The one very salient point, so far as Cornwall was concerned, was that it could be depended on for generations for the production of tin. The world depression at the present time had been brought about through dredges working on alluvial tin, mostly in Malay and Siam, and they knew that the life of those deposits was very short, compared with the mineral deposits in Cornwall. Remarks were sometimes heard from the older people that there were not so many mines in Cornwall as there used to be. Forty years ago there were a number of mines working at St. Just, for instance, but the amount of mineral produced by only two mines there the year before last was greater than any mineral production in the St. Just area in the whole of its history. Science had enabled mines to deal with larger tonnages.

The Society's gold medal for the year has been presented to Professor Lawn, president of the Institute of Mining and Metallurgy, for his work in mining geology.

### The Case for Nickel Coinage

THE case for nickel coinage, with a plea for its wider adoption in the British Empire, are set out in an article in a recent issue of the *Nickel Bulletin*.

"The chief virtues of copper-nickel money compared with bronze," the article states, "are its pleasing colour, its comparative resistance to corrosion, and its hardness. The whiteness of the metal is due to the nickel content, 25 per cent. of nickel serving to produce a pleasing white colour. The potency of nickel in this respect may easily be seen by comparing a 75·25 per cent. copper-nickel coin with a worn British silver coin, such as were issued between 1922 and 1927, for though these latter coins contain proportionately 25 per cent. less copper than the copper-nickel coins, their colour in the worn parts resembles that of straw. Superior as copper-nickel is to bronze as a coinage metal, it falls short in many ways to pure nickel, especially in regard to its liability to counterfeiting, its lesser durability, and its non-magnetic property. Seeing that nickel and nickel alloys are nowadays the most popular metals employed for token coinage throughout the greater part of the world, it seems strange that the British Empire, and particularly the Mother Country, should lag behind other countries in adopting them; the more so when it is borne in mind that 90 per cent. of the world's production of nickel comes from the Dominion of Canada.

### Replacement of Bronze

"THERE are good reasons perhaps why Great Britain, with her wide-flung interests, should be cautious in substituting nickel for silver in her silver coinage, for any step taken that contributes still further to the recent fall in the value of silver must necessarily reduce the purchasing power of the rank and file of the populations in the East; in such countries as instance as China, where the currency is still on a silver basis. But there seems to be no sound reason for hesitating to replace the cumbersome bronze pennies and half-pence of the Empire by clean copper-nickel coins of more convenient size and attractive appearance."

The writer goes on to give details of the nickel coinages adopted throughout the Empire from the first issue in Jamaica in 1869 to that of the Irish Free State two years ago, and concludes with a review of the Empire's position as a producer of coinage metals.

## Trade, Commerce, Finance : The Month in Review

### From Our Market Correspondent

THERE has been no improvement whatever during the past month in the iron and steel trade of this country, and we are now entering upon the last month of the year with no promise of its being any less gloomy than the period through which we have just passed. True, there is plenty of talk of an early change towards better trade. The Federation of British Industries has issued its forecast of an upward movement early in the New Year, a forecast which has been heartily endorsed by the Chancellor of the Exchequer in one of his recent speeches. One can only hope that they are true prophets, but even if they are we have still some dreary weeks ahead of us, under trading conditions which have been unparalleled for many years past. Some of the district reports have indicated rather more inquiry but there is nothing very tangible in that, and actual business shows no increase.

#### Continental Steel Prices

Prominence has been given to the advance in prices of Continental steel, but it would be a stretch of the imagination to describe that as a result of an improvement in trade. The disturbance which arose from the break-up of the European Steel Cartel is settling down, and this increase in prices is a natural consequence of that settlement, as it was obviously impossible for the ruinous prices at which foreign steel has been offered of late to continue indefinitely. The advance varies from 5s. to 7s. per ton, but, after all, what help is that to us? It has practically no effect on the position as far as this country is concerned. The prices quoted by Belgium, France and Germany for export, for plates and sections, are 50s. to £3 per ton below our prices, and it is obvious therefore that it will need several advances like that which has just been announced to remove the handicap under which our steel works are suffering; and nothing short of protection will achieve that result.

#### Foreign Dumping

This leads us to another consideration, which is worthy of attention. It is a point which seems to have been overlooked, although the Chairman of the South Durham Iron and Steel Co. dealt with it in his speech a few days ago. There has been a lot of talk in Parliament and in the press about the condition of the iron and steel industry, a lot of useless advice has been given by people who know little or nothing of the inside of the trade, and there has even been the threat of parliamentary action, presumably similar to that taken in the coal mining industry, to compel the steel makers to put their house in order. They are literally commanded to rationalise and re-organise.

The scope of such schemes is in the elimination of waste and overlapping, and the reduction of overhead charges. The best conceived of such schemes may possibly effect a saving of some shillings per ton in the cost of production, put it even as high as 10s. per ton which is quite an outside figure not likely to be attained. How far does that go to meet the position disclosed by the figures quoted above? If everyone worked for nothing we should still be unable to bring our costs below the continental selling prices. When that fact has got through to the understanding of those who are so ready to criticise the steel makers and whose parrot cry is "rationalisation!" they may begin to realise that the real remedy lies elsewhere. By all means let rationalisation be carried on, in the true meaning of the word, and not merely lumping together a lot of inefficient units; but at the same time steps must be taken to prevent the wholesale dumping of foreign steel into this country; without that, rationalisation is ineffective.

Given that protection, however, our steel works would enjoy a much larger share of our home trade, which would in turn give them more power to compete in the export markets. It must not be forgotten that the post-war growth of competition in the overseas markets, from the other steel producing countries, has become a serious menace to our own export trade; and if Russia succeeds in her five-year plan, as she may well do, we shall find it still harder to maintain a footing.

#### Misleading Reports on Steel Industry

It would really be doing a great service to the industry as a whole if we could get some authoritative report on its actual condition, compiled by someone who is actively engaged in it and who knows the inside working from practical experience, and have that report given wide publicity as a counter to the misleading reports that are flying about. It is true that there are some inefficient plants which might well be closed down, but on the whole our steel works are up to date and efficiently operated, bearing in mind the particular class of trade for which this country is best suited, which, let it be said, is not the mass production of the American kind. There is far too much readiness to condemn the steel trade as altogether incompetent, and it is annoying to find so much eager prominence given to adverse reports, while little or nothing favourable is published. It will be remembered that not long ago a Mr. Repleglo of America published a report on our iron and steel industry, which received wide publicity in the press. Of course, he condemned the industry as being hopelessly inefficient. Yet when inquiries were made, no one could find any connection between him and our steel industry which entitled him to make such a report. He was quite unknown to the leaders of the industry, and he had no inside knowledge of it, otherwise he would not have made the unpractical suggestions that he did. Now the politicians are taking up the tale and lecturing the steel makers on their shortcomings. It is high time they were shown what untiring efforts the steel makers have put forth and the measure of success they have achieved in trying to overcome the difficulties which were created by the war and the burdens imposed by the politicians since the war.

#### Price Levels

It is not safe to say that our home selling prices have yet reached bottom, and until, in the opinion of buyers, that point is reached, we are not likely to see any extensive buying movement. The manner in which the prices for uncontrolled materials are cut by the competing makers is sufficient to deter consumers.

Moreover it is none too certain that controlled prices will retain their present level. In pig iron a reduction of 2s. 6d. per ton all round was made in the middle of November by the Central Pig Iron Producers Association which controls the supply in the Midlands. This was done to meet the threatened Continental competition, and seems to have achieved its object, although it has not brought out much business. The Cleveland makers are firmly maintaining their prices, refusing to make any further concessions. They have steadily reduced prices and output to meet the falling off in demand, but have now reached a price level beyond which they will not go. The hematite makers are following the same policy, as they have reduced production almost to the minimum. As for the controlled prices of steel plates and sections, they must remain as they are until the next joint association meeting in January, but there will be little business done before then. An advance is, of course, out of the question, yet notwithstanding the depression in trade it is to be hoped that no reduction is made at that meeting, as it would bring no compensatory benefit to the steel makers. When the rebate and the carriage are deducted, these prices are very lean ones. The most pressing problem at present is to secure sufficient orders to keep the mills running. Short time working appears to be general throughout the trade. Shipbuilding is slack, constructional engineering is also quiet, while the locomotive builders and the boiler makers have less work than for some time past. The development of the use of steel arches and props for the collieries is helping those steel makers who have fitted themselves for this work, but the total weight is comparatively small.

The production figures for October show a further decrease. The pig iron output was 415,000 tons, compared with 425,000 tons in September. Eight furnaces were put out of blast during the month. The output of steel was 512,500 tons, compared with 580,600 tons in September.

**Some Inventions of the Month****By Our Patents Correspondent**

Abstracts of other Patents of metallurgical interest will be found in our Patent Literature published weekly in THE CHEMICAL AGE.

**Alloys for Pistons**

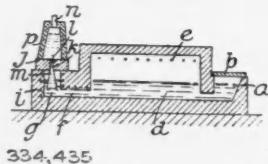
ALUMINIUM-SILICON alloys particularly suitable for the pistons of internal combustion engines are described in Specification 334,656, bearing the International Convention date December 22, 1928, by Aluminium, Ltd., of Toronto, Canada, Assignees of R. S. Archer and L. W. Kempf of Cleveland, Ohio, U.S.A., and in Specification 334,777, bearing the International Convention date August 15, 1929, by K. Schmidt Ges. of Neckarsulm, Wurtemburg, Germany.

334,656. The alloys contain about 7-15 per cent. of silicon together with 0.2-3 per cent. of magnesium, 0.5-7 per cent. of nickel, and 0.7-3 per cent. of copper. Manganese, chromium, or titanium may also be added, and iron may be present as impurity. The Specification is open to public inspection under Section 91 (3) (a) of the Acts contains additional subject-matter in that the silicon content of the alloys may be raised to about 25 per cent.

334,777. The alloys contain 70 per cent. or more of aluminium and about 15-25 per cent. of silicon, together with about 0.2-3 per cent. of chromium and 0.5-3 per cent. each of manganese, cobalt, and nickel, with or without 0.5-1 per cent. of titanium or molybdenum. Antimony may also be added to the extent of 0.5-3 per cent., and iron may be present in some cases.

**Casting Metals**

In making castings of aluminium or its alloys the moulds *p* rest on a detachable chilling plate *j* in front of a melting and degasifying furnace *d*, and the plate is provided with tubes *m* for introducing the metal into the moulds from the



furnace. Each tube is conical and is aligned with a double conical hole *k* in the plate. To fill the moulds the air is exhausted through tubes *n*. The furnace *d*, fitted with electric heating elements *e*, is supplied at *a* with molten metal from a main furnace. Cooling means may be provided near the constriction *l* to ensure solidification and ready fracture, and solidification may be controlled by means for heating the moulds locally or entirely. See Specification 334,435 bearing the International Convention date October 29, 1928, by Vereinigte Aluminium-Werke Akt.-Ges. of Lausitz, Germany.

**Electrolysis**

In the electrolytic deposition of gold and silver the electrolytes used are solutions of the oxygen-free iodine compounds of the metals in solutions of salts such as iodides of the alkali or alkaline earth metals. A current density of 100 amperes per square metre at 0.1-0.3 volts is suitable, and soluble or insoluble anodes may be used. See Specification 333,402, bearing the International Convention date December 11, 1928, by A. Mozer of Berlin.

**Reducing Ores**

In the reduction of iron or other ores with carbonaceous reducing agents in a shaft furnace, the combustible gases escaping from the upper part of the furnace are partly burnt with oxygen and blown back into the charge, a continuous circulation of the gases being thus maintained. Alternatively the escaping gases may be treated for removal of carbon dioxide, as by freezing or absorption, partly burnt with oxygen, passed through carbon or coal to increase their content of carbon monoxides, again partly burnt with a further supply of oxygen and blown back into the charge. The application to the production of iron sponge is described and illustrated in detail. The oxygen required may be obtained either by the Linde process or by electrolysis of water, and in the latter case, the hydrogen generated may be blown into the furnace. See Specification 334,500, dated, April 26, 1929,

by E. A. A. Grönwall and H. J. H. Nathorst, of Stockholm, Sweden.

**Titanium**

A PROCESS for producing homogeneous titanium or ferrotitanium melts by aluminic-thermal reaction is described in Specification 333,816, bearing the International Convention date November 1, 1928, by W. and H. Mathesius of Berlin. Chromates, tungstates, manganates, permanganates, uranates, and like salts of acids derived from the oxides of the heavy metals are added to the mixture to effect a regular deposition of the regulus. Such salts may be alkali or alkaline earth salts, or may be salts of heavy metals such as iron, nickel, cobalt, manganese, uranium, tungsten, molybdenum, or vanadium, in which case the metallic radicle passes, together with the metal from the acid radicle, into the titanum or ferro-titanium regulus.

**Welding Copper**

A WELDING rod for use in the acetylene or like gas process of welding copper is described in Specification 333,466, dated November 2, 1929, by W. Andrews, of Norton-on-Tees, Durham, and Imperial Chemical Industries, Ltd., of London. It consists of copper together with small quantities of silver and deoxidant, and is coated with a deoxidant and flux. Thus a copper alloy may be used containing 0.5 per cent. of silver and 0.06 per cent. of phosphorus coated with a mixture consisting of 1 part of fluorspar and 3 parts of a powdered deoxidant alloy containing 15 per cent. of magnesium, 30 per cent. of manganese, and 50 per cent. of silicon. A suitable deoxidising mixture in the coating consists of iron or copper with a high percentage of manganese, silicon, or phosphorus. An example is given of a rod comprising a copper alloy containing 0.06 per cent. of phosphorus silver plated with 0.5 per cent. of silver and having a coating consisting of 3 parts of ferro-silicon containing 80 per cent. of silicon, 2 parts of phosphor copper containing 15 per cent. of phosphorus, and 1 part of a mixture of equal parts of lime and felspar. The work may be coated with a flux comprising an aqueous paste of 6 parts of boric acid, 1 part of aluminium phosphate, and 1 part of fused borax.

**Commercial Intelligence**

The following are taken from printed reports, but we cannot be responsible for any errors that may occur.

**Mortgages and Charges**

[NOTE.—The Companies Consolidation Act of 1908 provides that every Mortgage or Charge, as described therein, shall be registered within 21 days after its creation, otherwise it shall be void against the liquidator and any creditor. The Act also provides that every Company shall, in making its Annual Summary, specify the total amount of debts due from the Company in respect of all Mortgages or Charges. The following Mortgages and Charges have been so registered. In each case the total debt, as specified in the last available Annual Summary, is also given—marked with an \*—followed by the date of the Summary, but such total may have been reduced.]

DOMINION STEEL CORPORATION, LTD., Sheffield.—Registered November 11, £400 debentures, part of £5,000 and 5 per cent. bonus payable in certain events; charged on part of building in Queen Street, Sheffield, also general charge. \*£1,000. August 4, 1930.

KAMUNTING TIN DREDGING, LTD., London, E.C.—Registered November 14, £100,000 debentures (secured by Trust Deed dated November 6, 1930); charged on mining leases in F.M.S., also general charge. \*Nil. January 2, 1930.

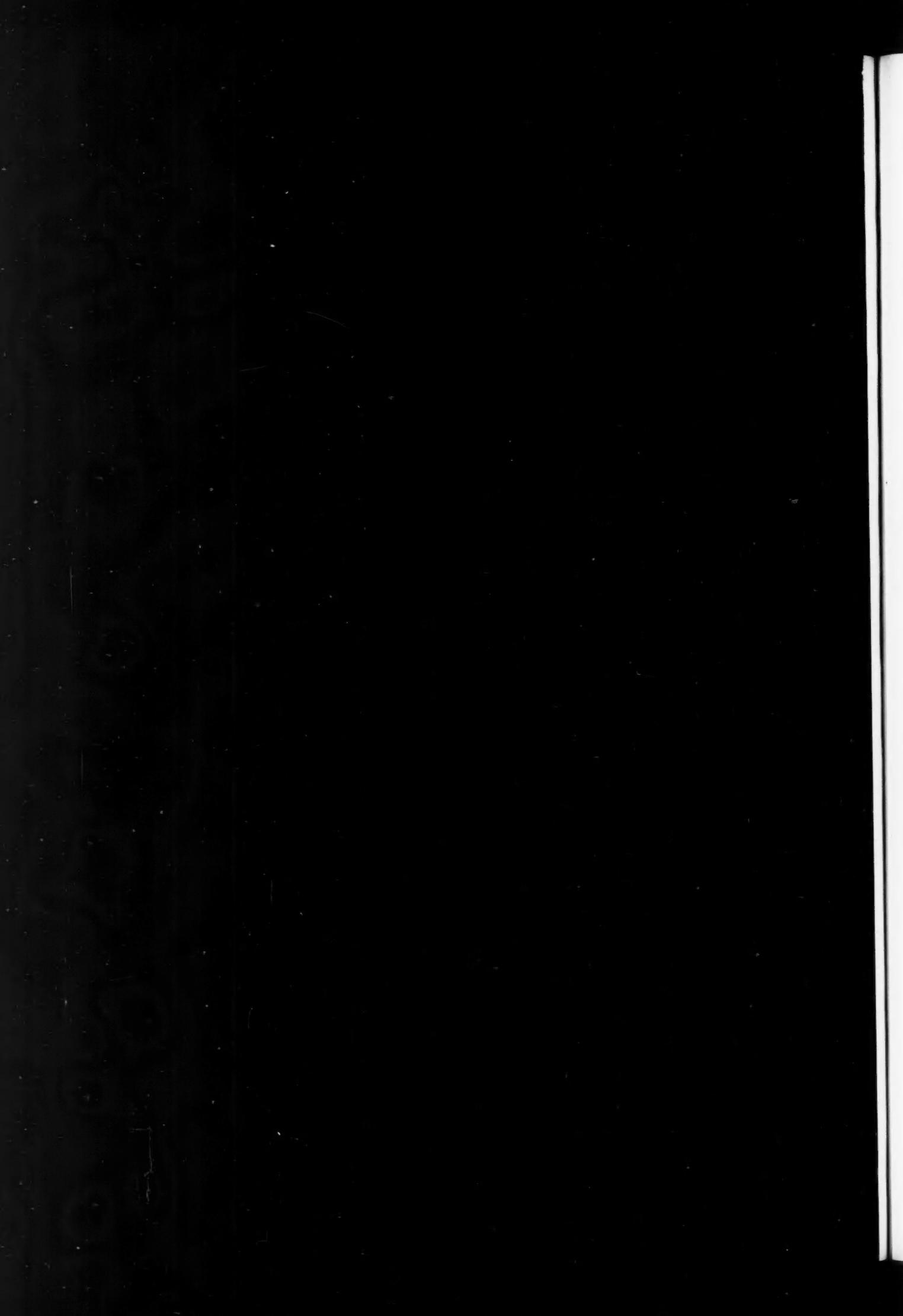
MERU TIN, LTD., London, E.C.—Registered October 30. \$42,857.14 collateral charge (supplemental to £5,000 debenture dated October 11, 1929), to Eastern Smelting Co., Ltd., Princes House, Gresham Street, E.C.; charged on a mining lease. \*£5,000. December 27, 1929.

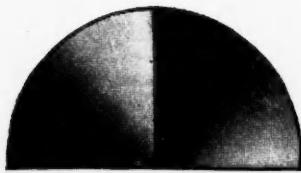
TAVOY TIN DREDGING CORPORATION, LTD., London, E.C.—Registered October 23. £125,000 (not ex.) debenture stock and 5 per cent. premium (secured by Trust Deed dated October 17, 1930); charged on certain mining leases, etc., also general charge. \*—. August 6, 1929.

**Satisfaction**

WELDLESS STEEL TUBE CO., LTD., Wednesfield.—Satisfaction registered October 29, £100,000 (not ex.), registered November 17, 1926.

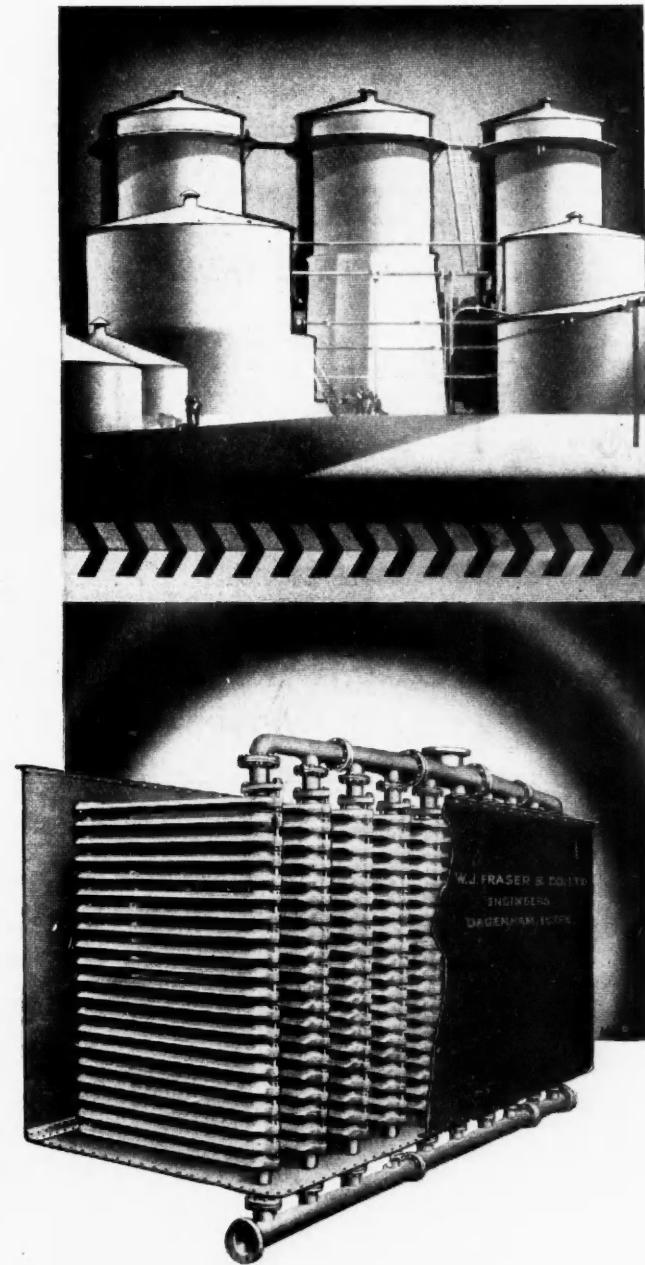






## MINERAL OIL REFINING PLANT

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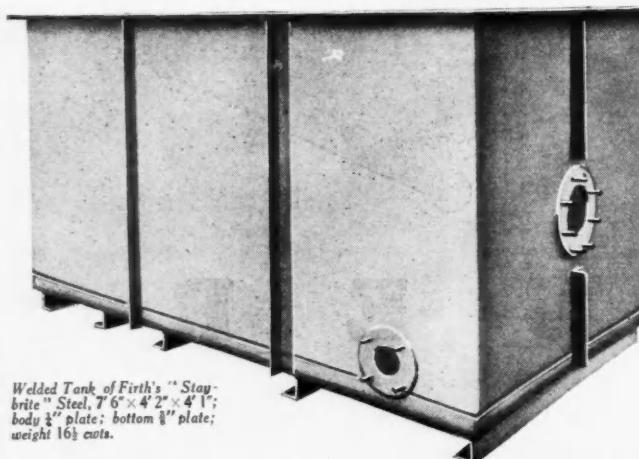
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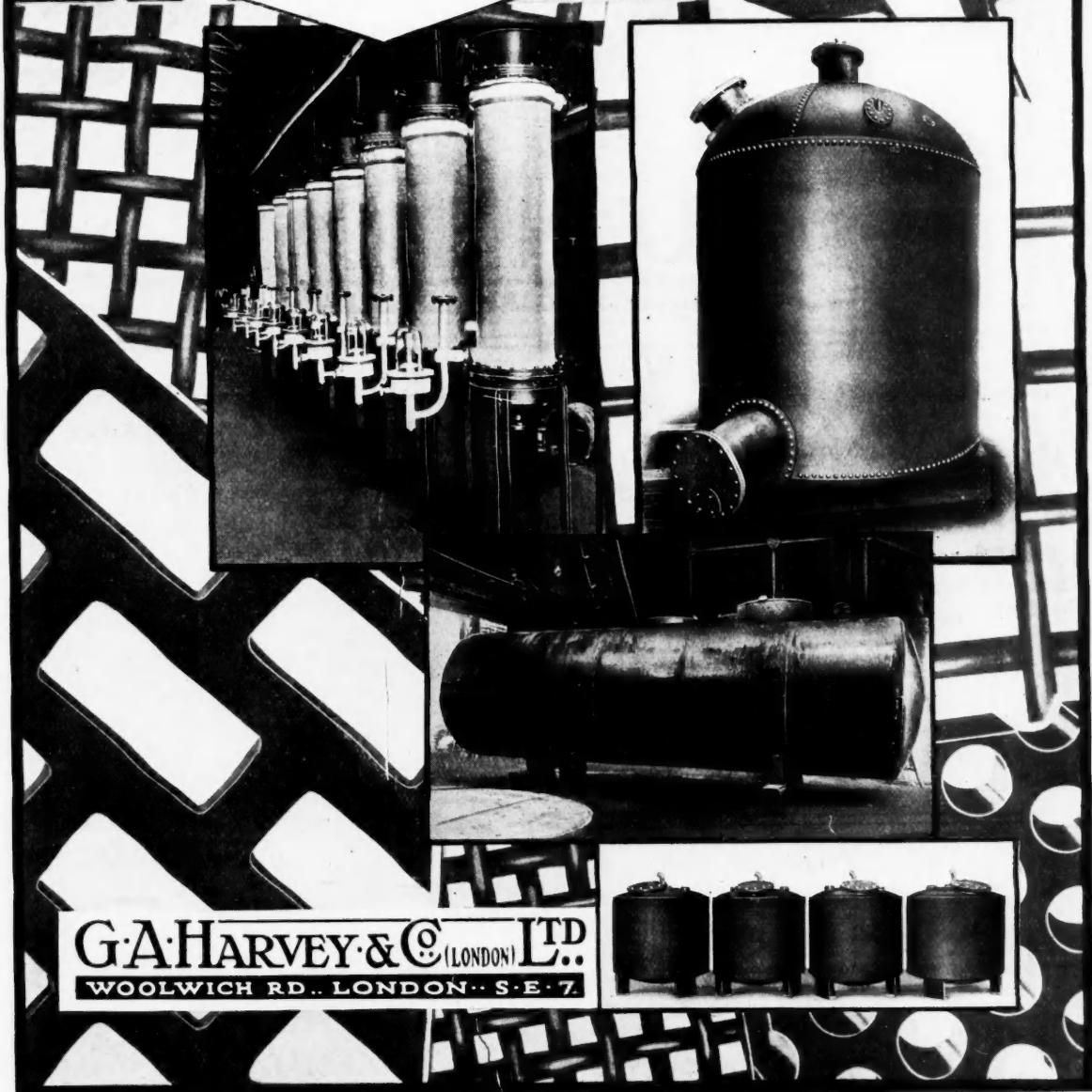
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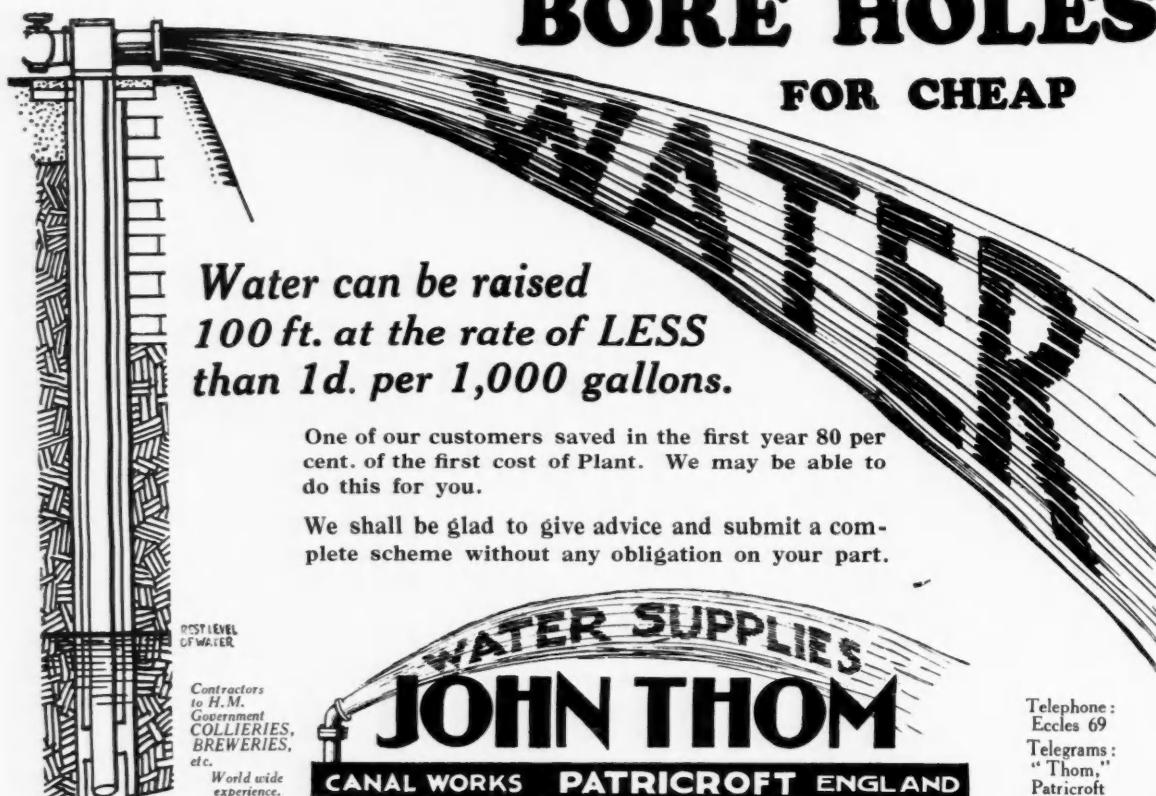
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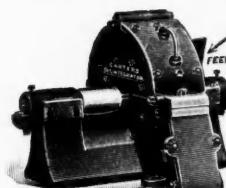
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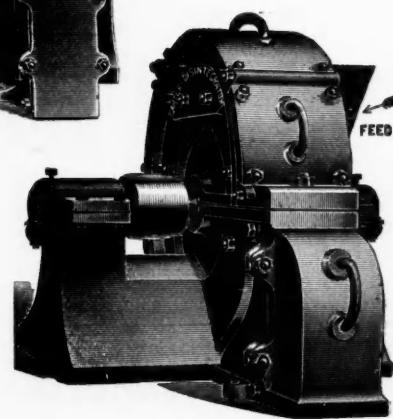
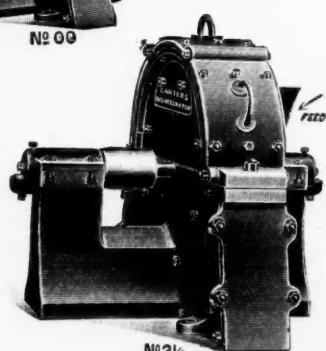
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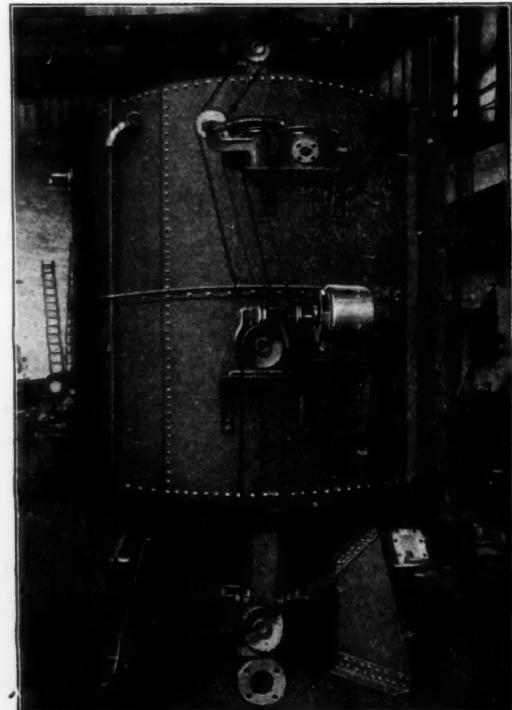
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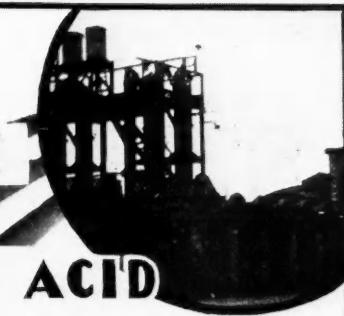
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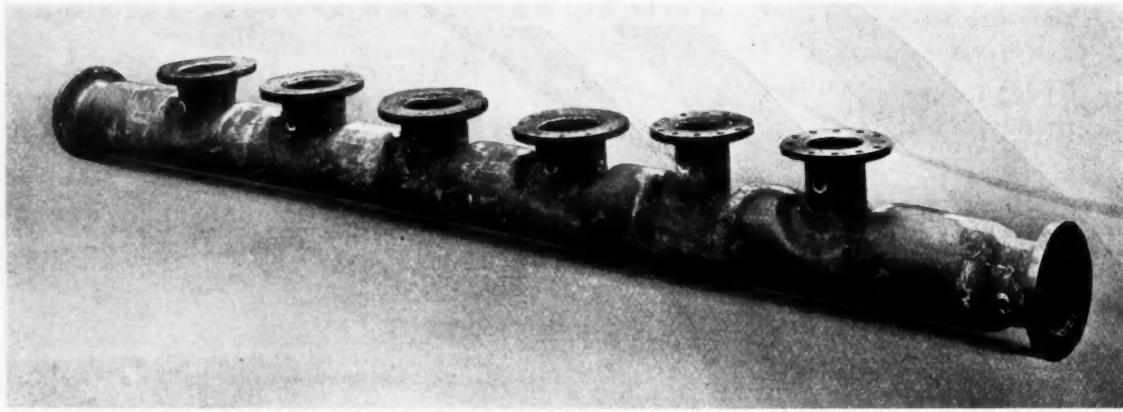


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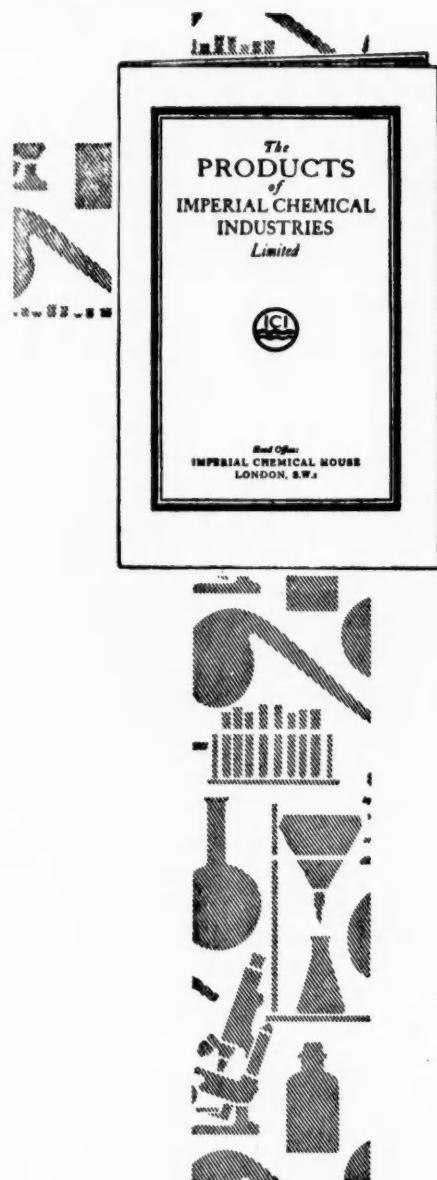
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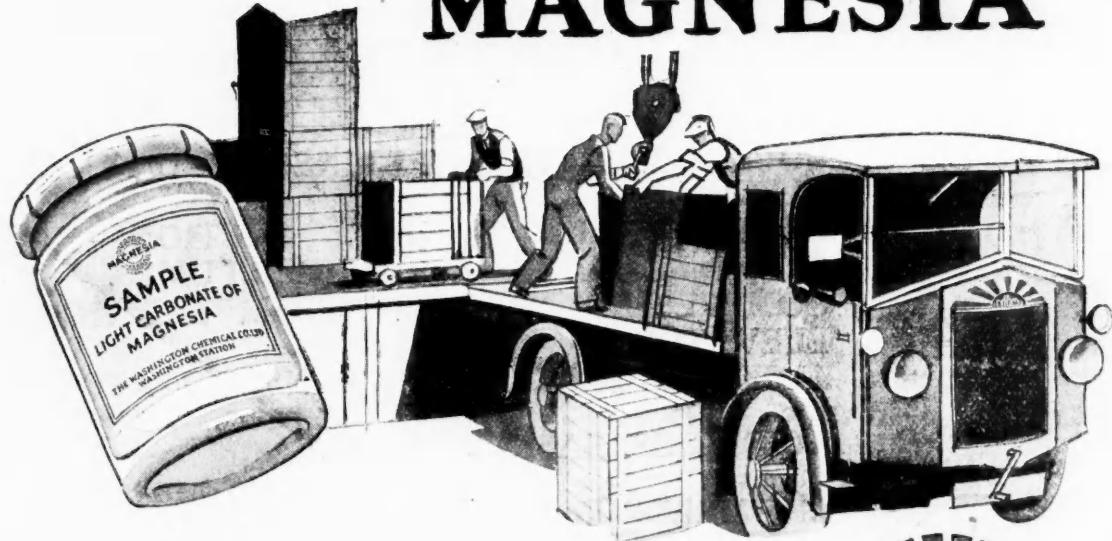
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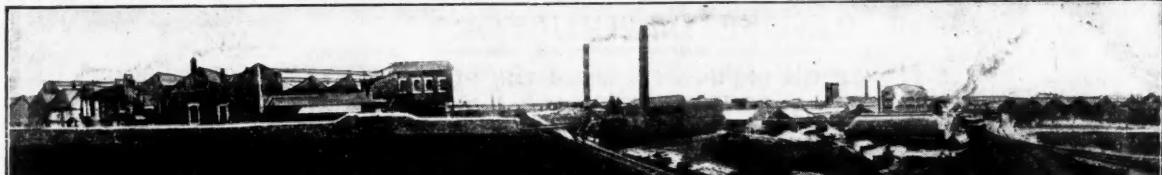
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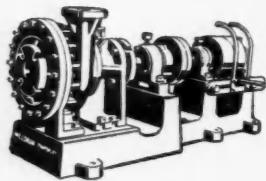
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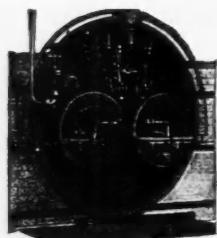


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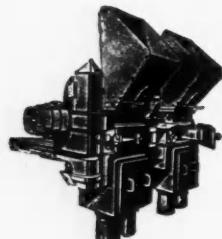
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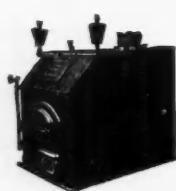
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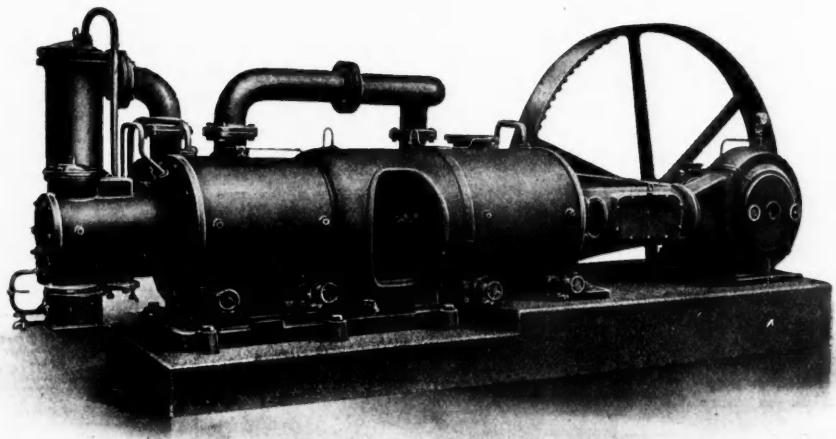
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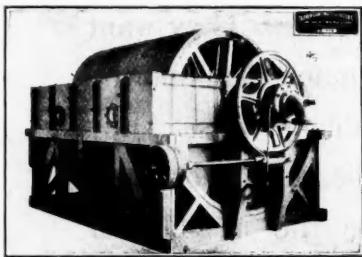
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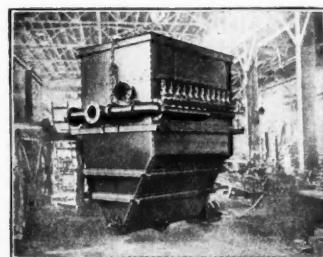
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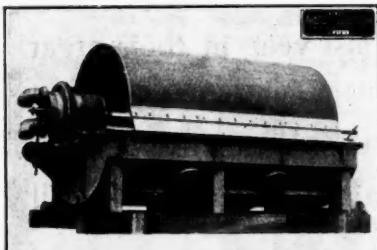


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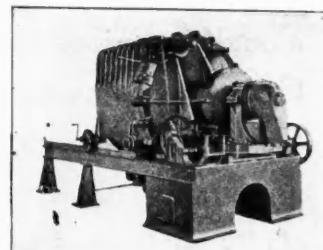


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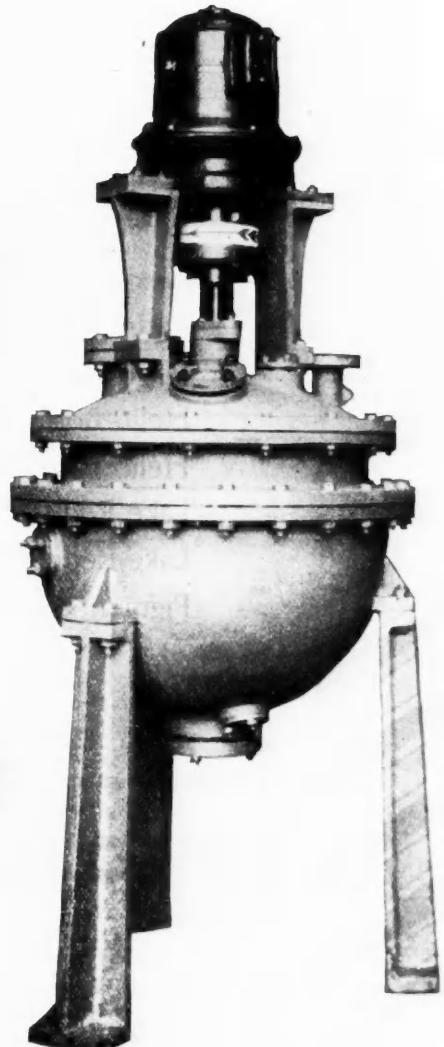
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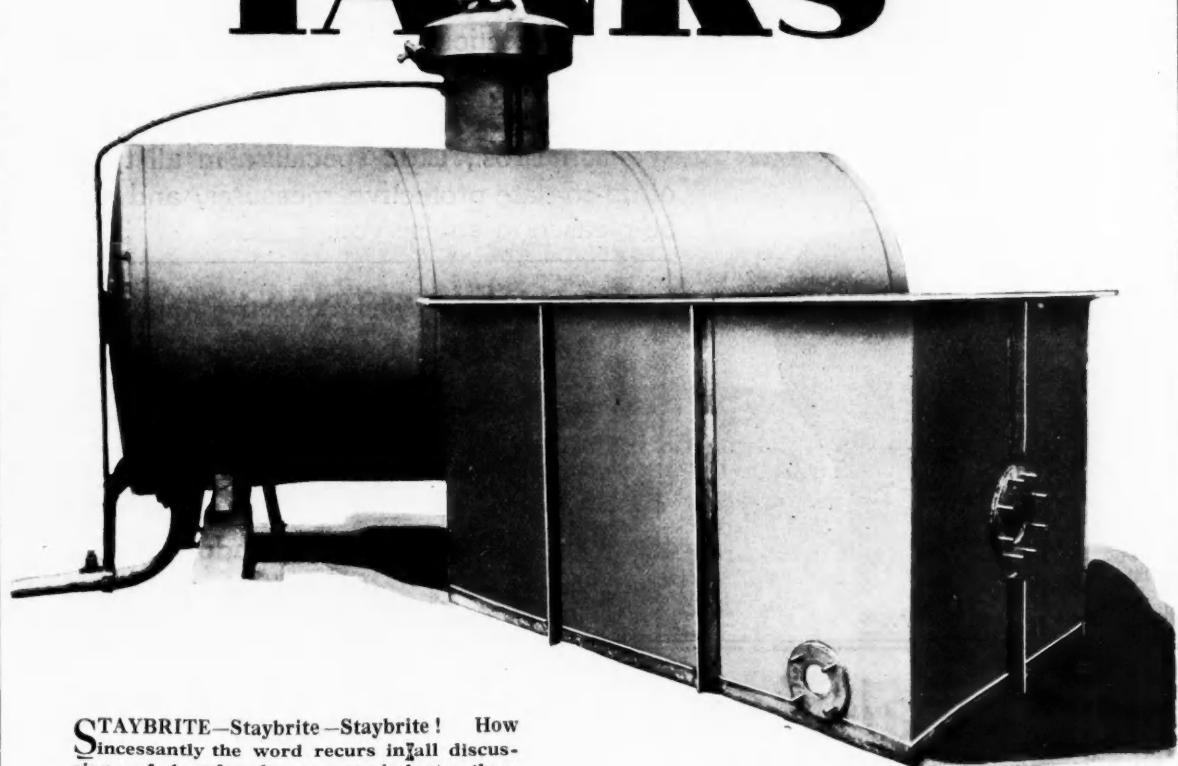
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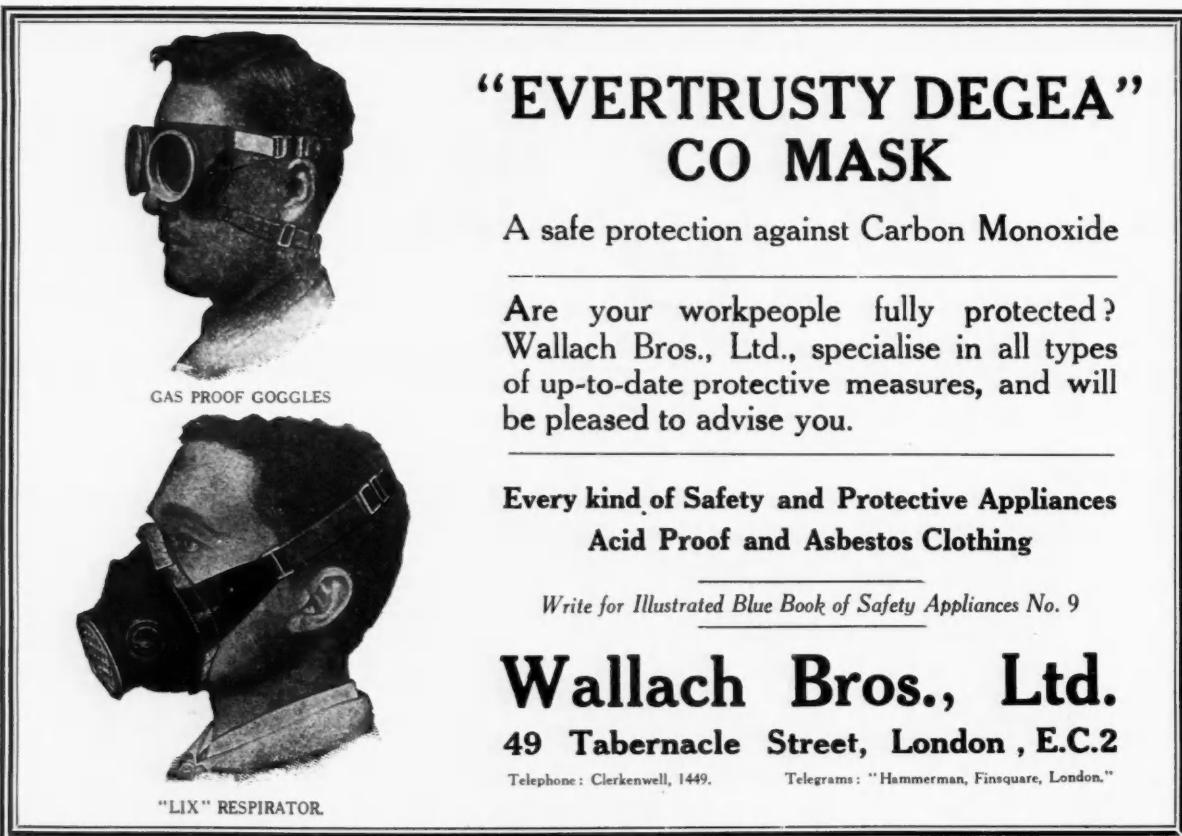
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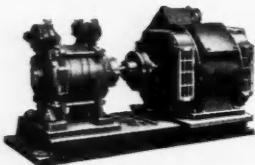


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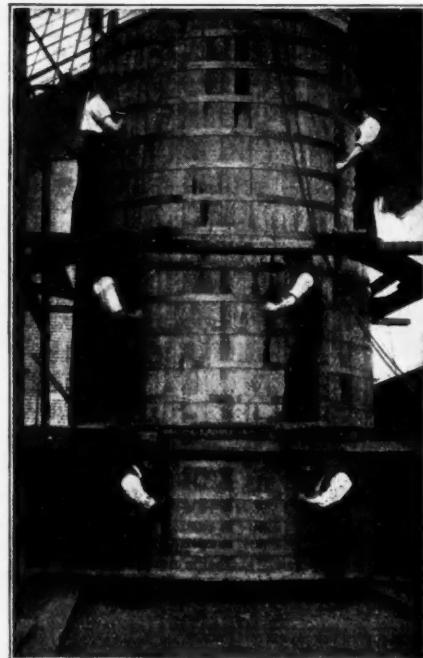
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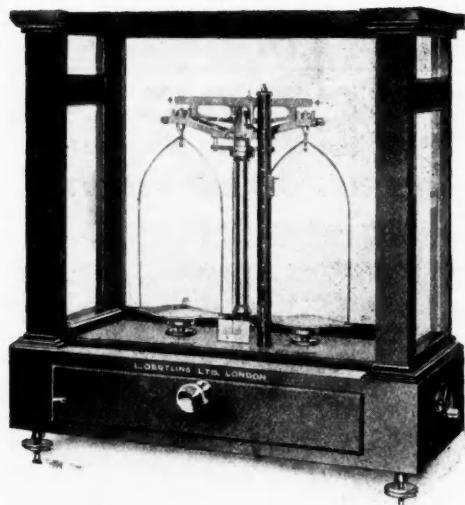
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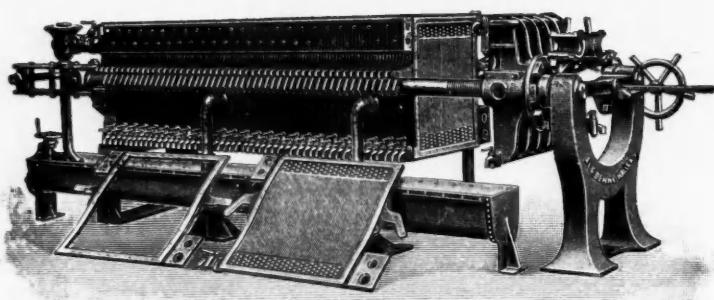
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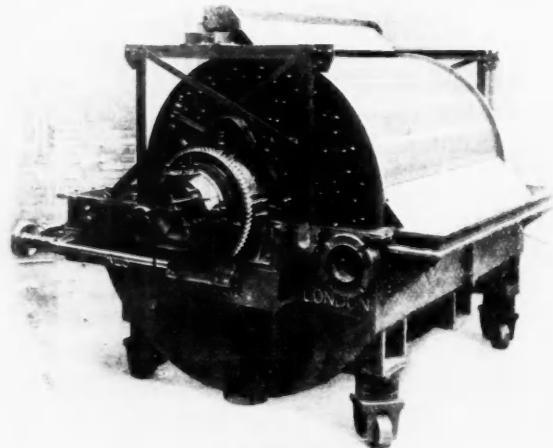
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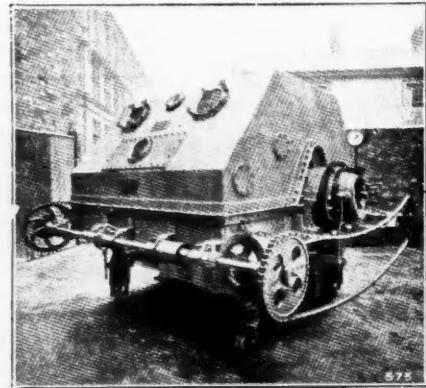
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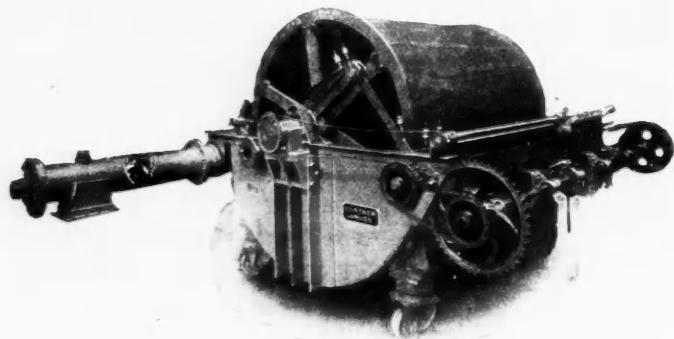
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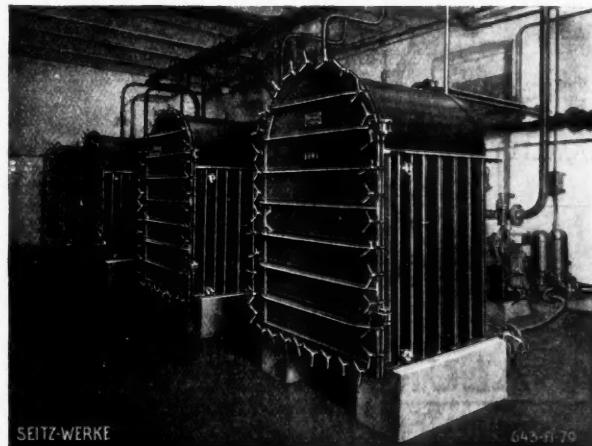
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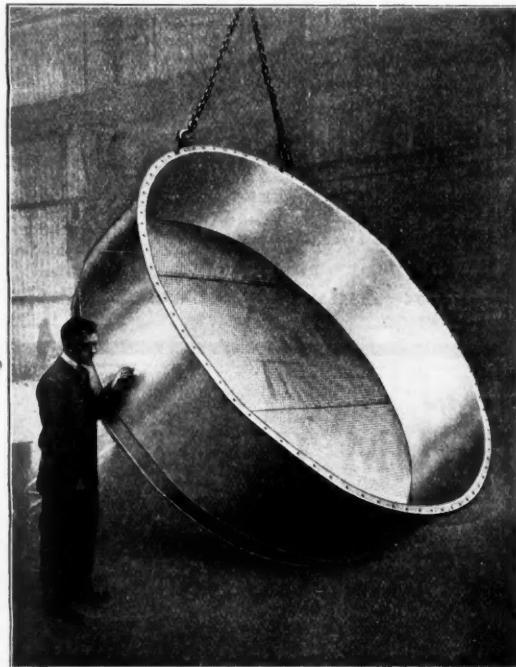
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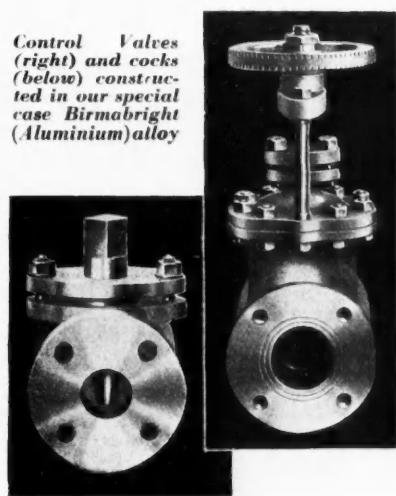
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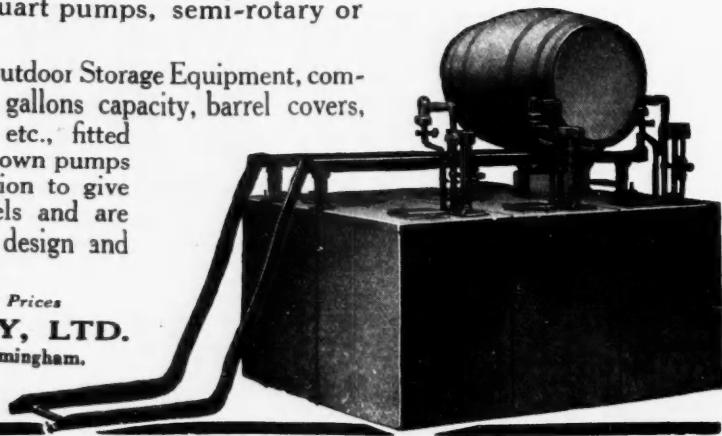
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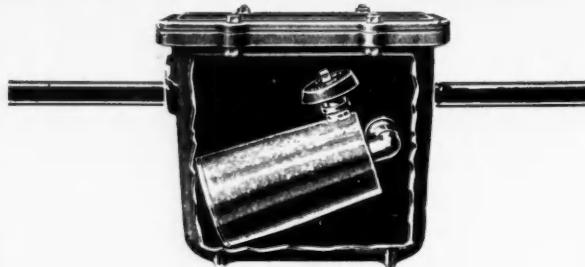
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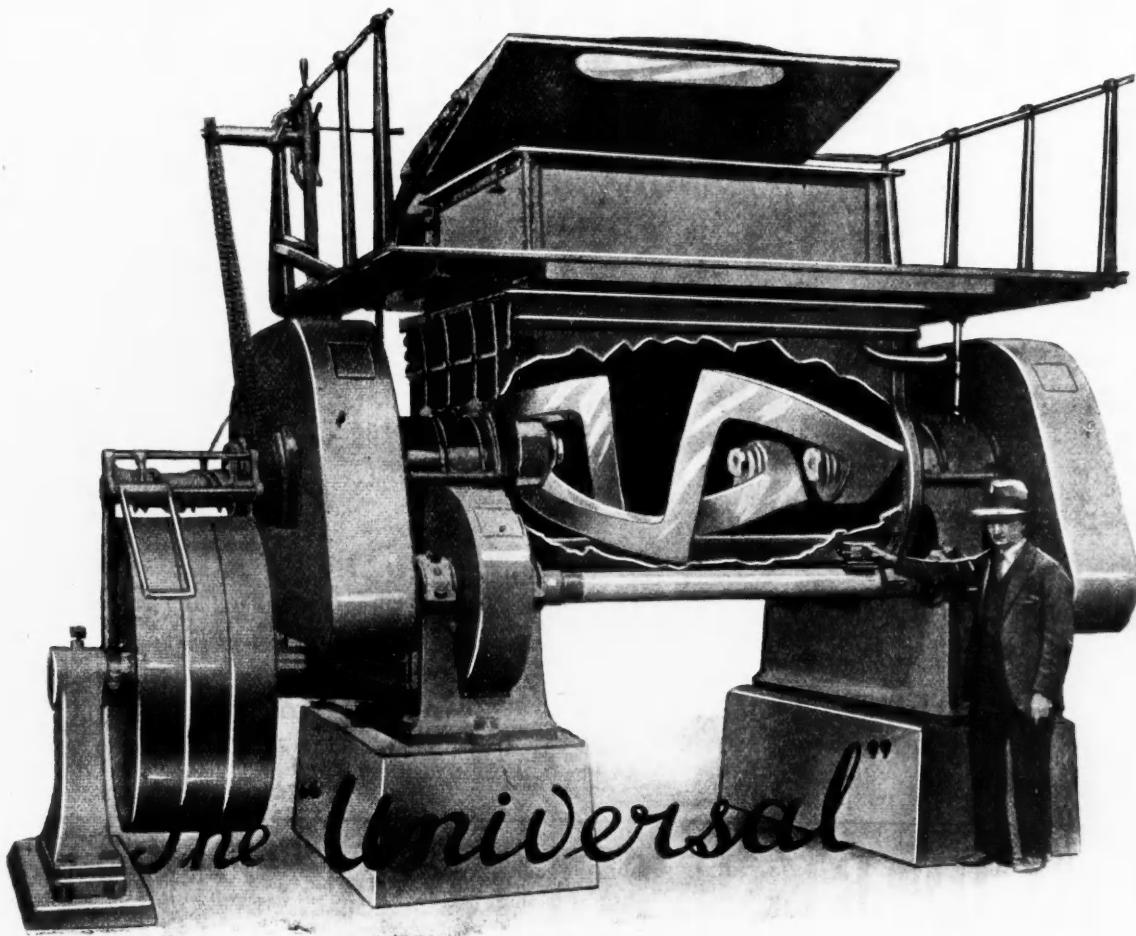
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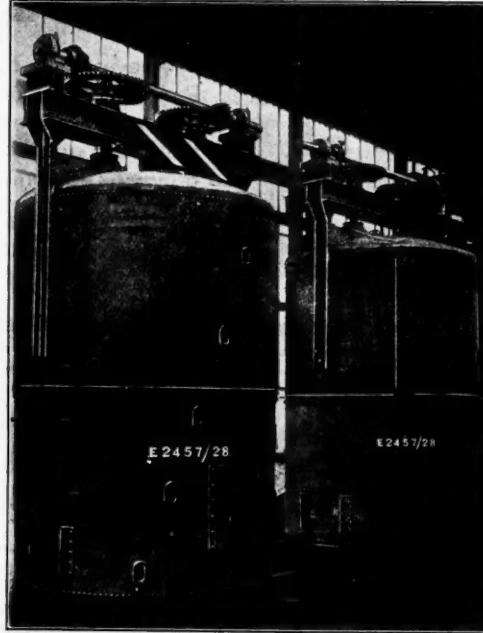
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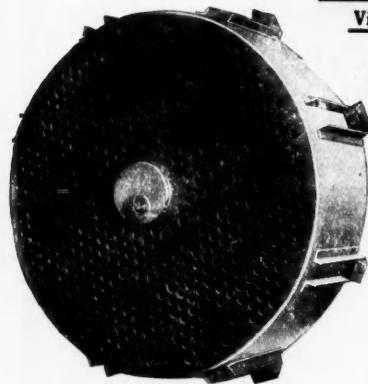
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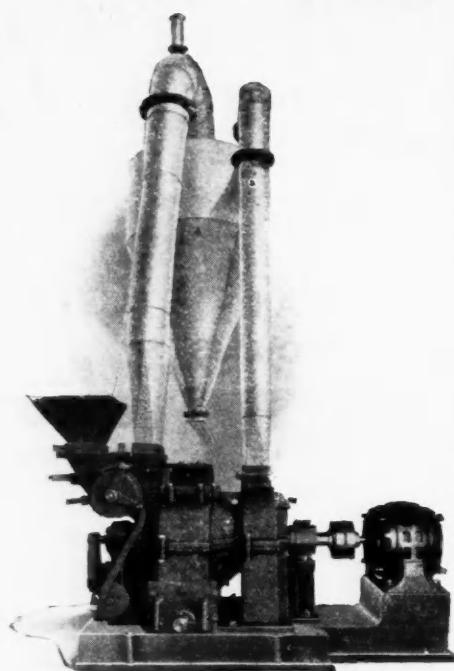


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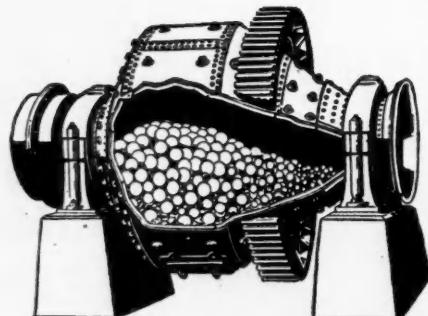
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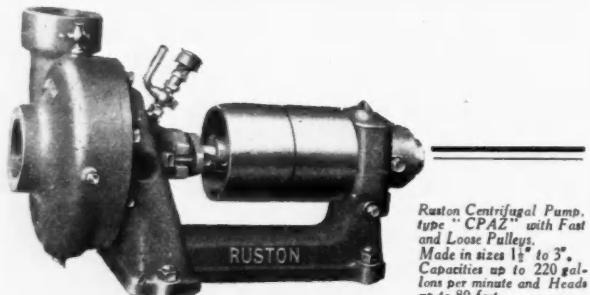
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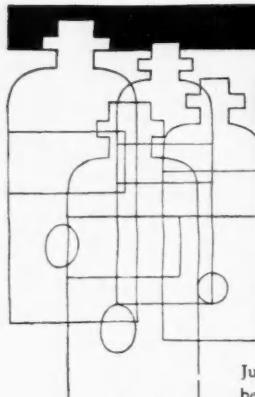
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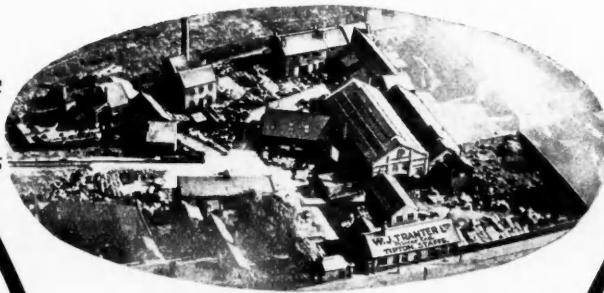
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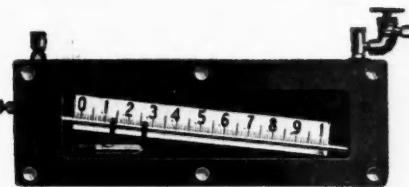
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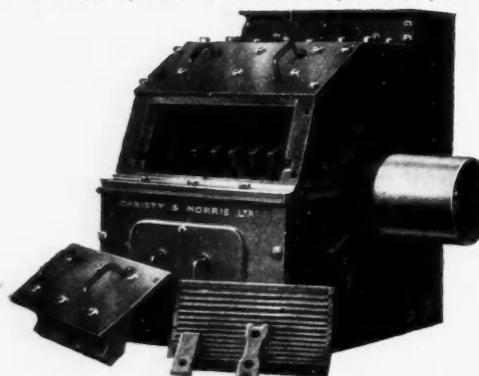
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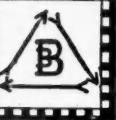
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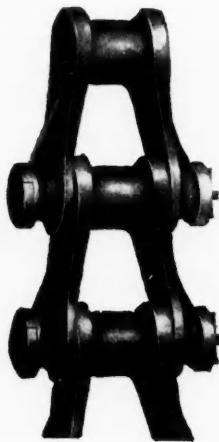
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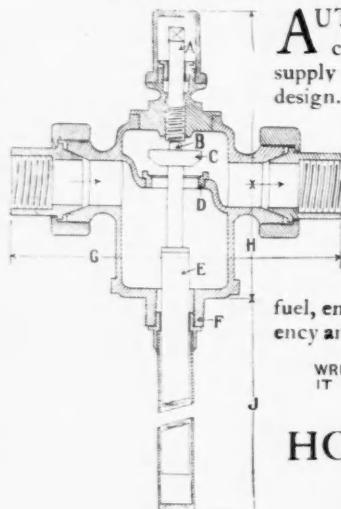
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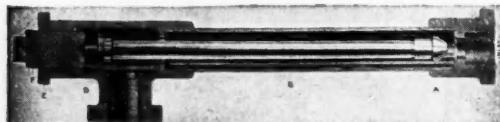


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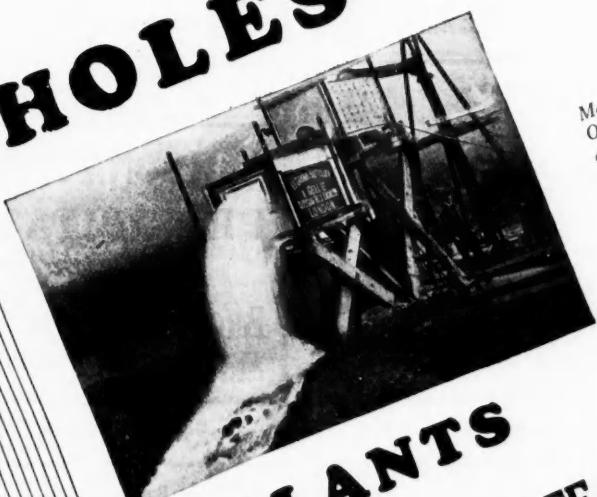
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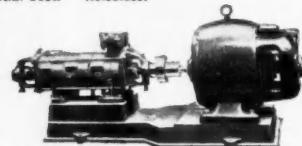
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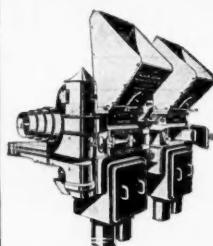
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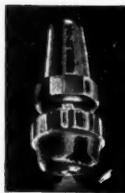
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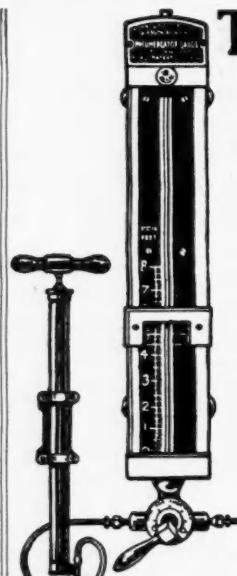
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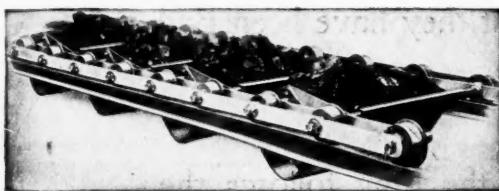
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